Laboratory Schedule

Week	Exp. #	Activity	Assignments Due*
Jan 19 – 23		*No labs this week*	Manual Quiz
Jan 26 – 30	1	Qualitative Analysis of Metal Cations	Exp. 1 Pre-lab Quiz
Feb 2 – 6	2	Molar Mass Determination via Freezing Point Depression	Exp. 2 Pre-lab Quiz Exp. 1 Report
Feb 9 – 13	3	Characterizing Reaction Kinetics	Exp. 3 Pre-lab Quiz Exp. 2 Report
Feb 16 – 20	4	Evaluating Chemical Equilibria	Exp. 4 Pre-lab Quiz Exp. 3 Report
Feb 23 – 27	5	Measuring Acid Dissociation Constants	Exp. 5 Pre-lab Quiz Exp. 4 Report
Mar 2 – 6	-	Missed lab (1-5) make-up	Exp. 5 Report Notebook Check 1
Mar 9 – 20		Spring Break	
Mar 23 – 27	6	Buffer Properties	Exp 6 Pre-lab Quiz
Mar 30 – Apr 3	7	Oxidation-Reduction and Electrochemistry	Exp. 7 Pre-lab Quiz Exp. 6 Report
Apr 6 – 10	8	Ester Synthesis	Exp. 8 Pre-lab Quiz Exp. 7 Report
Apr 13 –17	9	Ester Characterization	Exp. 9 Pre-lab Quiz Exp. 8 Report
Apr 20 – 24	10	Polymer Synthesis and Properties	Exp. 10 Pre-lab Quiz Exp. 9 Report
Apr 27 – May 1	-	Missed lab (6-10) make-up	Ester Summary Report
May 4	-	*No labs this week*	Exp. 10 Report Notebook Check 2

*Assignments are due at 11:59 PM the day your section meets each week, except pre-lab quizzes, which are due just before class

Grading and Assignments

The laboratory component is worth 20% of your overall CHM 104 grade. Your total points for laboratory work may be scaled up or down to reflect this. The following table details all graded work in CHM 104 Lab.

Assignment		Points
Manual Quiz		10
Pre-lab Quizzes	5 pts x 10	50
Reports	20 pts x 10	200
Ester Summary Report		40
Notebook Checks	25 pts x 2	50
Total		**350***

*Note that this value will be scaled to 20% of the overall points in CHM 104.

There are three main assignments for each experiment to be completed each week:

Pre-lab prep
In order to maximize time in the laboratory, you must prepare for each experiment before arriving to class. Pre-lab prep for each experiment consists of five things:

- Reading the manual chapter
- Answering the pre-lab questions (consult instructor if confused)
- Starting the notebook entry
- Completing the pre-lab quiz (use of your manual, your lab notebook, and a calculator is permitted, no internet or outside help allowed)

Notebook entry
Each experiment must have its own entry in your notebook. With every experiment, be sure you are following the notebook guidelines detailed on page vi. Your instructor will evaluate your notebook for completeness during the Notebook Checks.

Lab report
The report sheet for each experiment contains questions that evaluate your understanding of what happened during the experiment. You may choose to answer these questions in your notebook while in class, but the final report should be submitted as a .pdf file (see appendix D) on Moodle. The assignment folder in Moodle contains a .docx file that you can use to type your answers. Handwritten reports are permissible, as long as you photograph or scan them and convert to .pdf for uploading to Moodle. If you did not understand the pre-lab quiz, be sure to seek advice from your instructor before starting the report.

Preparing for Class in the Laboratory

You should come to the lab each week with:

- Lab-appropriate clothing and safety goggles (see page vii)
- Lab notebook
- Pen and calculator
- Previous lab report finished or mostly finished
- Pre-lab prep and quiz completed
- Notebook entry started (see next page and Appendix B)

Potential workflow of a successful student in a Tuesday CHM 104 Lab

Day	Activity
Mon	Start notebook entry for next lab and then complete pre-lab quiz
Tue	Attend lab section: Perform experiment, filling out lab notebook with procedure and observation notes After class: Begin lab report, noting any difficult questions
Wed	Finish most or all of report and read through next lab, noting any confusion with pre-lab questions.
Thurs	Contact instructor about any questions on report or pre-lab questions
Fri	Put finishing touches on report and submit to Moodle.
Sat	*Chill out and relax*
Sun	Review pre-lab questions.

It is inadvisable to wait until the night before class to do all the prior week's work.

Missing Class and Late Work

You should only miss your lab for illness or emergencies. If you are dealing with a crisis that requires you to miss class, please contact your dean.

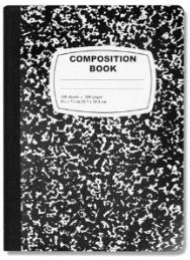
If you do miss class, it is your responsibility to contact your instructor (preferably before class) and arrange for a way to complete the experiment.

Reports submitted past the due date posted on Moodle will have 10% of the possible points deducted per day late. Late work will not have points deducted if you have been excused by a dean or at your instructor's discretion.

Quizzes will not be accepted late unless excused by a dean or instructor.

Maintaining a Laboratory Notebook

A laboratory notebook is your official record of what was done for each experiment. A good laboratory notebook should be written in such a way that someone of equivalent skill could use only your notebook and replicate exactly what was done and obtain the same results.



General guidelines:

- Use a bound composition notebook (see photo at right)
- Write only with non-running ink (no pencils or felt-tip markers e.g. Sharpies)
- Number every page
- Maintain a table of contents (start your first experiment a few pages in)
- Do not scribble, black out, or white out mistakes. Simply cross off the erroneous text with a single line and explain what the mistake was. Everything, even mistakes, should be legible.
- All data must be recorded in your notebook! Masses, observations, etc. must be recorded in your notebook, not your manual.

For each experiment:

- Starting on a new page, write the title of the experiment and date the entry
- Thoroughly read through the experiment in your manual. Write a purpose for the experiment, briefly explaining what you will do, make, or investigate.
- Write a balanced net ionic equation for any reactions performed
- Answer the pre-lab questions in your notebook
- Write out the procedure step-by-step. Do not copy the lab manual, write as if you are leaving instructions for yourself to follow.
    - This can be done before the experiment is performed as long as you leave room for observations and other data, as well as potential in-the-moment changes to the procedure
- Collect your quantitative data in a table to make calculations easier
- Show any relevant calculations clearly, including units and significant figures
    - Include chemical labels as well - don't just write "0.3 mol" - include moles of what! e.g. "0.3 mol NaCl"
- Print and tape any generated plots or draw them
- End your notebook entry with a conclusion that answers the question or provides the data that the purpose laid out.

2 Molar Mass Determination via Freezing Point Depression

Goals

- Determine the freezing point of stearic acid with a cooling curve
- Determine the freezing point of stearic acid with an unknown solute
- Determine the molar mass of the solute based on freezing point depression
- Utilize spreadsheet software (Excel) to construct data plots

Pre-lab Questions

1. Which is the colligative property: *freezing point* or *freezing point depression*?
2. What are the units of *molality*? This is not molarity (M).
3. What is ΔT_f if the freezing point of a pure water is 0 °C and the freezing point of a solution of salt water is -16 °C?
4. What is the intersection point of the following two lines:

   $$y = 2x + 4 \text{ and } y = 3x + 1$$
5. How many moles of solute are in a solution of 340 g solvent if the molality was determined to be 0.55 mol/kg? Assume the van 't Hoff factor is 1. Use what you know about the units of molality.

- To plot your data, bring a computer to class.

Background

Solutions are homogeneous mixtures that contain two or more substances. The major component is called the *solvent*, while the minor component is called the *solute*. Saltwater is a solution, with water as the solvent and salt as the solute. Since the solution is primarily composed of solvent, physical properties of a solution primarily resemble those of the solvent. However, some properties can be influenced by the solute. These *colligative* properties are dependent only on the concentration of the solute and not on the identity or other properties of the solute.

Colligative properties arise because solute particles essentially get in the way of solvent molecules. The solute can prevent solvent molecules from interacting with each other. One colligative property is *freezing point depression*. When a substance freezes, many particles come together, often in an ordered manner. If solute particles are around, they prevent this coming together, to an extent. When salt is added to water, the salt particles prevent water molecules from coming together to form the ordered crystal structure of ice. This is why salt is distributed on roads to prevent ice - the salt dissolves in the water and prevents it from sticking together, effectively lowering the freezing point.

Other colligative properties include boiling point elevation, lowering of vapor pressure, and osmotic pressure.

We can quantify how much a solute depresses the freezing point of a substance. The freezing point of a solution differs from that of the pure solvent by an amount that is directly proportional to the concentration of the solute particles. This relationship is shown in Equation 1.

$$\Delta T_f = iK_f m \quad (1)$$

Term	Definition
ΔT_f	Freezing point depression, or how much the freezing point has been lowered with the addition of solute
i	van 't Hoff factor, the number of particles a solute produces when dissolved (typically 1 for non-ionic compounds)
K_f	Cryoscopic constant of solvent, unique to each substance
m	Molality of solute, expressed as mol solute/kg solvent

Notice the unit of concentration used here is *molality,* not molarity. Molarity depends on the volume of a solution, which changes with temperature. Since we are dealing with variable temperatures in this experiment, we use molality, a unit of concentration that is not influenced by temperature. The unit for molality is moles of solute divided by kg of solvent.

In this experiment we will measure the freezing point depression of stearic acid with an unknown solute. To do this we must measure the freezing point of pure stearic acid and the freezing point of the mixture.

$$\Delta T_f = T_f(\text{pure}) - T_f(\text{mixture}) \quad (2)$$

Since it can be difficult to determine precisely when a liquid has turned into a solid, we will utilize temperature vs. time plots called *cooling curves* to reveal the freezing points.

For our experiments today:

- The van 't Hoff factor, *i,* for all unknowns is 1.
- The cryoscopic constant, K_f, of stearic acid is 4.50 °C•kg/mol

Procedure (Pairs)

1. Tare a large empty test tube on a balance.
2. Fill the tube with ~9 g of stearic acid and record the exact mass.
3. Heat a water bath to ~90 °C.
4. Immerse the tube in the water bath so that the stearic acid melts. Place a thermometer in the tube and ensure the stearic acid reaches 85 °C.
5. Keeping the thermometer in the tube, remove the tube and dry the outside with a paper towel.
6. Immediately record the initial temperature ("t0").
7. Record the temperature of the sample every 30 seconds. Stir constantly (gently) with the thermometer. Record these measurements in your notebook. If there is time between readings, you can also record it on a computer to save time during analysis. One partner can hold the tube and call off readings while the other records the data.
8. Keep collecting temperature readings until the temperature changes by less than 0.1 °C between readings. At that point, collect 6 more temperature readings.
9. Add ~1 g of an unknown sample to the tube and stearic acid. Record the exact mass added and which unknown was used. Do not weigh out more stearic acid, just use what is already in the tube.
10. Repeat steps 4-8 with this unknown mixture. Switch roles with your partner.

Cleanup

1. Remelt the contents of the tube and pour them into the disposal container.
2. Partially fill the tube with 2-propanol and gently heat it, stirring with the thermometer. Once dissolved pour this into the secondary disposal container. This step may need to be repeated until the tube is clean.
3. A scrub brush and dish soap should be used to clean. Your tube should have no trace of fatty acids when you are done.
4. You should end with a 2-propanol rinse so that your tube can dry out for the next class.

Analysis

1. Generate a plot of your data for the pure stearic acid and the unknown mixture. Follow the instructions in Appendix A to plot each set of data as two separate series. The first series should include all points up until the temperature starts changing by more than 0.1 °C per 30 seconds (the steep part of the curve). the other series should include the rest of the points (the flatter part of the curve). *Note: the time values should be entered as seconds (2.5 min = 150 sec).*

2. Fit a trendline for each data series and display the equations on the plot. The intersection of the two lines is the freezing point.

3. To find the intersection of the two lines, assume the two y values in the equations are the same and substitute one of the equations for y in the other equation. Since temperature is on the y-axis, the freezing point is the y value of the intersection point.
   a. If two lines have the equations $y = mx + b$ and $y = nx + c$ and we assume y for each line is the same, then $mx + b = nx + c$ and we can solve for x. To find the y value of the intersection point, plug the x value found into either of the equations and solve for y.

4. After calculating the freezing point for the pure stearic acid and the mixture, calculate the ΔT_f using Equation 2.

5. Calculate the molality of the mixture using Equation 1.
   a. Along with the information in the Background section, you should now know every value in Equation 1 except for molality.

6. Find the moles of unknown you added. You know molality is moles of solute divided by kg of solvent. The solvent is stearic acid.

7. Calculate the molecular weight of the unknown solute, using the grams you weighed out and the moles you just calculated.

8. Make sure your temperature readings are in your notebook before leaving.

3 Characterizing Reaction Kinetics

Goals

- Describe how initial concentrations of reactants affect reaction rates
- Use the initial rate method to determine the rate of a reaction
- Determine the kinetic order of reactants in a reaction
- Determine the rate constant for a reaction

Pre-lab Questions

1. If rate is expressed in units of M/s and concentrations in units of M, determine the units on the rate constant of this reaction: Rate = $k[A]^2[B]^1$
2. A rate law for a certain reaction is expressed as Rate = $k[A]^2[B]^0$. The rate of this reaction is found to be 5.0×10^{-3} M/s when [A] = 0.10 M and [B] = 0.10 M. Find the rate constant.
3. 35 mL of a 0.40 M solution of sodium thiosulfate is added to a beaker with 45 mL of water. 15 mL of 2.5 M KCl is added. What is the final concentration of thiosulfate in the beaker?

- To plot your data, bring a computer to class.

Background

Chemical reactions occur at varying speeds. They can range from very slow to extremely fast. For example, the rusting of iron is slow, whereas the explosive decomposition of TNT is extremely fast. We refer to the study of the rates of reactions as *chemical kinetics*. Reactions can only happen when reactants collide with each other with the proper amount of energy and in the proper orientation. Thus, the rates of homogeneous reactions in solution depend upon:

- The nature of the reactants
  - Large molecules react more slowly than small molecules because their size prevents access to the reaction site. Oppositely charged ions will find each other and react more rapidly than neutral molecules.
- The concentration of the reactants
  - Reactions take place when molecules collide. The chance of a collision is higher if there are more molecules around.
- The temperature
  - Heat adds energy to the system, causing particles to move faster. Faster moving molecules collide with more molecules in a given amount of time than slower moving molecules.
- The presence of a catalyst

- Catalysts speed up reactions by either bringing reactants together in the correct geometric arrangement for the reaction to occur or by providing a lower energy pathway for the reaction to take.

Kinetic experiments typically yield information about the speed or *rate* of the reaction and the *order* of the reaction. Consider the hypothetical reaction

$$A + B \rightarrow C + D \quad (1)$$

We can express the overall rate of this reaction in terms of the concentrations of reactants as a *rate law*:

$$\text{Rate} = k[A]^m[B]^n \quad (2)$$

The brackets around A and B denote concentration, *k* is the *rate constant* unique to each reaction, and the superscript *m* and *n* denote the extent to which the concentration of each species influences the rate (note: these terms have no relation to the stoichiometric coefficients of the reactants).

The rate of a reaction can be described in units of molarity per second, M/s. The concentrations of reactants A and B are expressed in molarity, M. The units of the rate constant *k* will depend on the order of each reactant - *m* and *n.* The overall order is equal to the sum of each reactant order - in this case, *m* + *n.*

The order exponents will determine the units for *k*. If *m* = 2 then there is a M^2 term that must be accounted for. The units for *k* must cancel out the units from the other terms to obtain a concentration over time unit such as M/s. If one of the reactant orders is 0, that term is equal to 1 and its units are discarded, since that reactant does not influence the rate. Thus, only the terms with non-zero exponents are considered when determining units.

In this experiment we will characterize the kinetics of a reaction and calculate *m, n,* and *k.* To do this we must measure the rate of the reaction with several different concentrations of reactants.

To find *m* and *n* we can measure the reaction rate while keeping the concentration of one reactant constant while varying the concentration of the other reactant - in this way we are only observing the effect of one reactant on the rate. Once *m* and *n* are known, *k* can be calculated.

The reaction we will characterize is the acid decomposition of thiosulfate:

$$Na_2S_2O_3(aq) + 2HCl(aq) \rightarrow S(s) + SO_2(g) + H_2O(l) + 2NaCl(aq) \quad (3)$$

This can be simplified to the net ionic equation:

$$S_2O_3^{2-}(aq) + 2H^+(aq) \rightarrow S(s) + SO_2(g) + H_2O(l) \quad (4)$$

The rate law for this reaction can be expressed as:

$$\text{Rate} = k[S_2O_3^{2-}]^m[H^+]^n \quad (5)$$

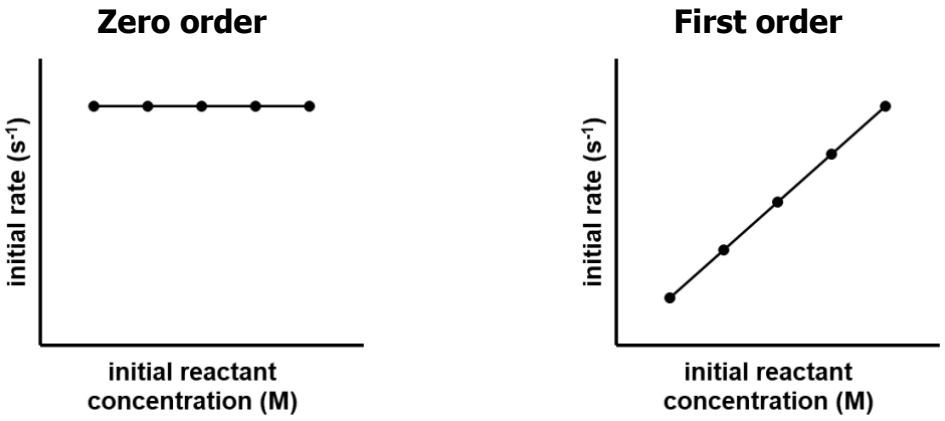
In one experiment, we can run several reactions, varying the initial concentration of $S_2O_3^{2-}$ while holding the initial concentration of HCl constant to approximate the rate law as:

$$\text{Rate} = k[S_2O_3^{2-}]^m \quad (6)$$

In a second experiment, we can do the same but vice versa: if initial HCl concentration is varied and initial thiosulfate concentration is held constant then:

$$\text{Rate} = k[H^+]^n \quad (7)$$

These two experiments will allow us to determine *m* and *n* by observing what sort of effect the initial concentrations have on the rate. The rate can be approximated as 1/units of time, or s^{-1}. Plotting this against the initial concentration of the reactant that is varied may yield a plot like one of the following:



In the zero order plot, we can see that the initial rate has no dependence on the initial concentration of the reactant. Thus the exponent in the rate law (*m* or *n*) would be 0. In the first order plot, we can see that the initial rate has a directly proportional dependence on the initial concentration of the reactant. Thus the exponent in the rate law would be 1.

In this reaction, we will directly control the initial reactant concentration. For the initial rate, we must use a defined completion point for the reaction. Since a solid precipitates in this reaction, we can take advantage of this visual cue to consistently determine an endpoint for each rate determination.

Procedure (Pairs)

Part A: Thiosulfate concentration dependence, determination of *m*

1. Place a ~150-mL beaker on top of a piece of paper with a dark black X on it.
2. Perform nine reaction trials by using the table below - add the HCl last, stir, and begin timing as soon as it is added using a stopwatch or clock. 5-mL and 50-mL graduated cylinders should be used for adding solutions to the flask.
3. Once the precipitate obscures the black X, record the time in your notebook.
4. Rinse out the beaker and complete the other trials. Be consistent in how you determine the endpoint.

Trial	Volume of 0.25 M thiosulfate (mL)	Volume of water (mL)	Volume of 2.0 M HCl (mL)	Total volume (mL)	
1	50	0	5	55	*15.22*
2	45	5	5	55	*15.38*
3	40	10	5	55	*16.79*
4	35	15	5	55	*19.43*
5	30	20	5	55	*22.00*
6	25	25	5	55	*26.67*
7	20	30	5	55	*34.72*
8	15	35	5	55	*46.22*
9	10	40	5	55	*80:02*

Part B: Acid concentration dependence, determination of *n*

1. Rinse out the 150-mL beaker and graduated cylinders.
2. Place the 150-mL beaker on top of a piece of paper with a dark black X on it.
3. Perform five reaction trials using the table below. As before, add the HCl last, stir, and begin timing immediately.
4. Once the precipitate obscures the black X, record the time in your notebook.

Trial	Volume of 0.25 M thiosulfate (mL)	Volume of water (mL)	Volume of 2.0 M HCl (mL)	Total volume (mL)	
1	20	30	20	70	*33.73*
2	20	34	16	70	*32.31sp*
3	20	38	12	70	*34.03*
4	20	42	8	70	*40.31*
5	20	46	4	70	*45.16*

Disposal

1. Solutions can be poured down the drain, rinsed with plenty of water.
2. Solid sulfur precipitate left on the glass can be wiped with a paper towel and placed in the trash.

Analysis

In order to determine the orders of each reactant (*m* and *n*) and the rate constant (*k*) you must compare the *initial rate* of each reaction to the *initial concentration of the reactant* whose concentration was varied. Both of these values must be calculated from your data.

1. As stated in the background, the initial rate here can be approximated as 1/time. Simply divide 1 by each time reading to obtain the initial rate with units s^{-1}. For example, for a reading of 5 seconds, the initial rate is 1/(5 seconds), or 0.2 s^{-1}.
2. To obtain the initial concentration of each reactant, use the dilution equation,
   $C_1V_1 = C_2V_2$. You know the concentration of the stock solutions (0.25 M thiosulfate and 2.0 M HCl), the volume of each of those you used, and the volume of the total solution.
3. These calculations can be done by hand or in a spreadsheet. Either way, put the initial rate and initial concentration data in a spreadsheet and plot them for each experiment (Appendix A may help).
4. Compare the plots to those shown in the background section and determine the order of each reactant (*m* and *n*).
5. Once the orders are known, you can plug them into the rate law (Equation 5) along with the concentration and rate data from any trial to determine the rate constant *k*. For this calculation, assume the units for the initial rate are M/s rather than just s^{-1}. You may have to do an additional dilution calculation to find the concentration of the other reactant. The units for *k* will result from the unit cancellations as you do your computation.

A spreadsheet with data from part A might look something like this:

	A	B	C	D
1	volume thiosulfate solution (mL)	time (s)	$[S_2O_3^{2-}]$	initial rate (s^{-1})
2	50	5	=(A2*0.25)/55	0.2

In this image cell C2 contains a formula that will automatically calculate the initial concentration of thiosulfate in the reaction solution (it is simply using $C_1V_1 = C_2V_2$). To generate the plot column D would be plotted against column C.

4 Evaluating Chemical Equilibria

Goals

- Understand that many reactions occur in both the forward direction and the reverse direction simultaneously, and when the rates of forward and reverse reaction are equivalent the system is in a state of equilibrium
- Understand that chemical systems respond to stresses to restore equilibrium according to Le Châtelier's Principle
- Observe changes in equilibrium based on chemical or physical stresses
- Predict changes in equilibrium concentrations of reagents based on stresses

Pre-Lab Questions

1. Copper (I) chloride is slightly soluble in water ($CuCl(s) \rightleftharpoons Cu^+ + Cl^-$). Suggest a substance that could be added that would reduce the equilibrium concentration of copper ion.
2. Considering Equation 4, identify a reagent (other than acetic acid) that could be added to shift the equilibrium to the right.
3. If a chemical reaction is endothermic, predict which direction the equilibrium will shift if the temperature is decreased.

Background

Some chemical reactions proceed until, for all practical purposes, one of the reactants is consumed completely. For example, consider what happens when gaseous HCl is dissolved in water. Recall that HCl is considered a "strong" acid. By definition, strong acids dissociate completely into ions in solution:

$$HCl(g) \rightarrow H^+(aq) + Cl^-(aq) \qquad (1)$$

Once formed, the ions do not recombine under ordinary circumstances to form covalent molecules. The reaction is said to "go to completion".

Contrast this with the behavior acetic acid ($HC_2H_3O_2$), a weak acid. When acetic acid is dissolved in water, *some* of the molecules ionize to form H^+ and $C_2H_3O_2^-$:

$$HC_2H_3O_2(aq) \rightarrow H^+(aq) + C_2H_3O_2^-(aq) \qquad (2)$$

However, the attractive forces between the H^+ and $C_2H_3O_2^-$ ions are strong enough so that some of the ions will recombine to form the molecular acid once again:

$$H^+(aq) + C_2H_3O_2^-(aq) \rightarrow HC_2H_3O_2(aq) \qquad (3)$$

Both of these reactions are happening simultaneously, with some ions separating and some combining. We can combine Equations 2 and 3 with a new symbol, two single-barbed arrows pointing different directions:

$$HC_2H_3O_2(aq) \rightleftharpoons H^+(aq) + C_2H_3O_2^-(aq) \quad (4)$$

This reaction is said to be reversible. We have arbitrarily chosen to put acetic acid on the left side of the equation, so its dissociation is considered the forward reaction, and the recombination of ions to form acetic acid is considered the reverse reaction.

When the forward and reverse reactions proceed at the same rate, the system is described as being at **equilibrium**. Both the dissociation and recombination of ions are happening at the same rate, meaning the concentrations of all three species remain constant. Note that the concentrations for these three species are not necessarily equal to each other, they simply remain constant over time.

All reversible reactions eventually reach a state of equilibrium. When the equilibrium is exposed to a **stress** (changes in reagent concentration, pressure, volume, or temperature), the system will respond to the stress in some way to restore equilibrium. This phenomenon is known as **Le Châtelier's Principle**. Consider the following generic equation:

$$A + B \rightleftharpoons C + D \quad (5)$$

At equilibrium, each species is present at a certain concentration. Both the forward and reverse reactions are happening simultaneously. If we add more of reagent C to the system, there are more molecules of C able to participate in the reverse reaction, so the rate of the reverse reaction increases, producing more of reagent A and B. The rate of the forward reaction eventually increases until it is equal to that of the reverse reaction, but the result is a net increase in the equilibrium concentrations of A and B, to counteract the increase in concentration of C.

When a stress results in an increase of concentration of reagents on the reactant side of the equation, it is said that the equilibrium has **shifted left** or that the **reverse reaction is favored**. Likewise, if we added B to the system, the equilibrium concentrations of C and D would increase, and we would say the equilibrium has **shifted right** or that the **forward reaction is favored**.

The removal of a reagent from the system can also shift the equilibrium, in the opposite way that adding a reagent does.

Changing the temperature can also effect changes in the equilibrium. Most chemical reactions are accompanied by a change in heat content, or enthalpy. We can consider the heat absorbed or given off by a reaction as being another reactant or product:

$$N_2(g) + 3\ H_2(g) \quad \rightleftharpoons \quad 2\ NH_3(g) + 92\ kJ\ heat \qquad (6)$$

When a reaction produces heat, it is considered **exothermic**. If it consumes heat, the reaction is **endothermic**.

Using Le Châtelier's Principle, we can predict the effect of temperature on the equilibrium position of Equation 6. For this reaction, raising the temperature (i.e., adding heat to the system) will shift the equilibrium to the left, while lowering the temperature (removing heat from the system) will have the opposite effect.

Le Châtelier's Principle can help us predict many shifts in equilibrium concentrations, depending on what is added or removed from a reaction. For example, if a gas is generated during a reaction that is conducted in an open container, the gas may evolve out of solution and be effectively removed from the system, causing a shift in equilibrium.

In today's experiment we will qualitatively observe the change in equilibrium when certain stresses are added to a system. Our reactions have been carefully chosen to provide a quick visual indication of the equilibrium shifts, and we can use what we know about equilibrium and Le Châtelier's Principle to rationalize why each change happens.

Procedure (Individual)

Be sure to note any changes to the appearance of the solutions as you complete the procedure.

Part A: Chromate - Dichromate Equilibrium

The yellow chromate ion (CrO_4^{2-}) is converted to dichromate ($Cr_2O_7^{2-}$) in the presence of acid.

$$2\ CrO_4^{2-}(aq) + 2\ H^+(aq) \rightleftharpoons Cr_2O_7^{2-}(aq) + H_2O(l) \qquad (7)$$

1. Pour 2 mL of the yellow 1 M potassium chromate solution into a test tube and add 3 M sulfuric acid dropwise with stirring until a noticeable change occurs.
2. Add several drops of 10% sodium hydroxide solution. Be sure to mix the contents of the test tube. Continue adding sodium hydroxide until a noticeable change occurs.
3. Discard chromates in the labeled hazardous waste container.

Part B: Cobalt (II) Complexes

The cobalt (II) cation, Co^{2+}, exists in water as pink complex, $Co(H_2O)_6^{2+}$. Cobalt (II) ions can form other complexes that have different colors.

$$Co(H_2O)_6^{2+}(aq) + 4\ Cl^-(aq) \rightleftharpoons CoCl_4^{2-}(aq) + 6\ H_2O(l) \qquad (8)$$

1. Pour the pink 0.2 M cobalt aqua complex, $Co(H_2O)_6^{2+}$, a chloride solution, into four test tubes, 2 mL each.
2. To the first, add 2 mL of 3 M HCl; to the second, 2 mL of 6 M HCl; to the third, 2 mL of 9 M HCl; and to the fourth, 2 mL of 12 M HCl. Mix well. Label the tubes if you think you may mix them up.
3. Place the test tubes in a boiling water bath for several minutes and note the results.
4. Put the test tubes in an ice water bath and note any changes in the appearance of the solutions.
5. Discard cobalt solutions in the labeled hazardous waste container.

Part C: Acetic Acid Dissociation

Consider the ionization of acetic acid:

$$HC_2H_3O_2(aq) \rightleftharpoons H^+(aq) + C_2H_3O_2^-(aq) \quad (9)$$

Since none of the species involved in this reaction are colored, we will use methyl orange, an indicator sensitive to the H^+ ion concentration. When a solution has a fairly high H^+ concentration, the indicator is **red**. At relatively low H^+ concentration, the indicator is yellow-orange.

1. Pour 2 or 3 mL of 0.1 M acetic acid into a test tube and add 2-3 drops of methyl orange.
2. Add a small quantity of solid sodium acetate ($NaC_2H_3O_2$) to the test tube and stir the mixture to dissolve the solid. Continue adding solid until a noticeable change occurs.
3. Add several drops of glacial acetic acid and mix well. Continue adding the glacial acetic acid until a noticeable change occurs.
4. Empty the tubes into the disposal container.

Part D: Thiocyano-Iron (III) Complex

Iron(III) ions react with thiocyanate to form the deep red thiocyano-iron (III) ion.

$$Fe^{3+}(aq) + SCN^-(aq) \rightleftharpoons FeSCN^{2+}(aq) \quad (10)$$

1. If there is already a beaker of $FeSCN^{2+}$ made, skip to step 4.
2. Transfer 1 mL of 0.1 M iron(III) nitrate to a 150 mL beaker, add 1 mL of 0.1 M potassium thiocyanate solution and pour it into the beaker.
3. Dilute the mixture with about 75 mL of distilled water to reduce the intensity of the deep red color and mix well.
4. Fill three test tubes about half-full with this solution. The first tube will serve as a color reference.
5. To the second tube, add 2 mL of 0.1 M iron(III) nitrate and mix the solution. Continue adding until a noticeable change occurs.
6. To the third tube, add 10 drops of 10 % sodium hydroxide solution and stir. Wait a minute for a change to occur, then if no change occurs add more NaOH.
7. Solutions in test tubes can be disposed of down the drain.

5 Measuring Acid Dissociation Constants

Goals

- Utilize a pH meter to generate a titration curve of an unknown acid
- Determine an equivalence and half-equivalence point of an acid-base reaction
- Calculate the dissociation constant of an acid-base reaction

Pre-lab Questions

1. Define equivalence point and half-equivalence point.
2. What is the pH of a solution with an H^+ concentration of 0.44 M?
3. What is the H^+ concentration of a solution with a pH of 4.54?
4. How many moles of base were used to titrate an acid if 23.45 mL were used to reach the equivalence point? The concentration of the base is 0.1449 M.

- To plot your data, bring a computer to class.

Background

Acid-base reactions involve the transfer of a proton between molecules. In strong acids, this transfer is total - nearly every molecule of acid has transferred a proton to the base. In weak acids, the situation is more complicated. To get an idea of what proportion of protons have been transferred, we must consider the equilibrium of the following reaction:

$$HA + H_2O(l) \rightleftharpoons A^-(aq) + H_3O^+(aq) \quad (1)$$

We can use this equation to describe the dissociation of the acid HA. You can think of the dissociation as the removal of the proton and subsequent generation of conjugate base A^-. The whole process can be described with a dissociation constant, K_a.

$$K_a = \frac{[A^-][H^+]}{[HA]} \quad (2)$$

In Equation 2 we have simplified H_3O^+ to H^+ and have removed water, since it is present in great excess. Before we move on, recall how pH is defined:

$$pH = -\log([H^+]) \quad (3)$$

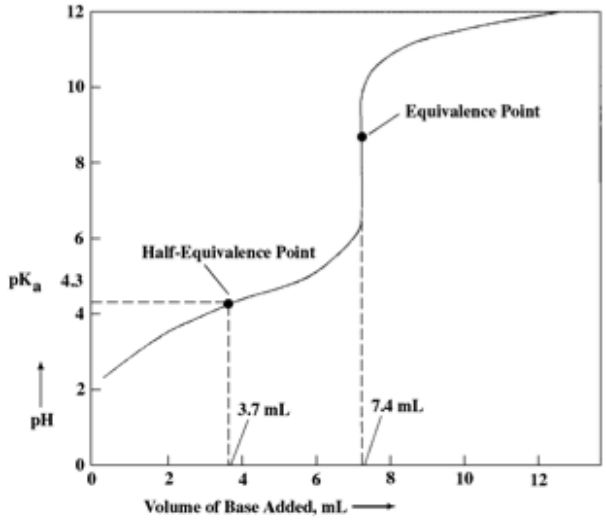
To take the "p" of something, one simply computes the -log of that something. Similarly we can compute the "p" of our K_a term to get pK_a:

$$pK_a = -\log\left(\frac{[A^-][H^+]}{[HA]}\right) \quad (4)$$

Notice both pH and pK_a contain -log($[H^+]$) terms. Using algebraic manipulation along with the properties of logarithms, we can obtain this equation:

$$pH = pK_a + \log\left(\frac{[A^-]}{[HA]}\right) \quad (5)$$

Equation 5 is known as the *Henderson-Hasselbalch equation.* It can help us determine K_a if we know pH, which is a property we can measure. If we titrate an acid with a strong base, we can see the pH of the solution increase. This is often displayed as a pH vs. volume of base added plot:



On this plot two points are of particular interest. The first is where the moles of added base are equivalent to the moles of acid. This is the *equivalence point*. In the titration experiment in CHM 103, this was the point at which the solution turned pink. The equivalence point is easy to find since the curve is nearly vertical at this point. It is the inflection point of the curve, or the spot where the curve changes from concave to convex.

The second important point is the *half-equivalence point*, defined as the point at which there is half as much base as acid. This point can be found by dividing the equivalence point's x coordinate in half and finding what y value intersects the curve at that x-value. At this point, half the acid has been titrated and half remains as HA. As a result of this, $[HA] = [A^-]$. Looking at Equation 5, and realizing that $\log(1) = 0$, we can see that at the half equivalence point, $pH = pK_a$! We can read the pK_a value by finding the y-coordinate at that point, then calculate K_a by reversing the logarithmic function of p:

$$K_a = 10^{-pKa} \quad (6)$$

In this experiment, you will generate a titration curve for an unknown acid and attempt to identify it by calculating the dissociation constant.

Procedure (Individual)

Titrations

1. Using a volumetric pipette, transfer 25 mL of unknown acid solution into a 250-mL beaker. Record which unknown was used.
2. Immerse the pH meter electrode in the solution and record the pH. Add enough distilled water to ensure the tip of the electrode is submerged.
3. Fill your burette to 0.00 mL with the NaOH solution. The concentration of NaOH will be provided by your instructor, make sure you write down what it is!
4. Add ~1 mL of NaOH solution to the flask and mix well. Record the pH as well as the exact volume added.
5. Keep adding ~1 mL of NaOH solution, mixing and recording the pH and exact volume each time.
    a. Unlike the titrations from CHM 103, there is no need to do any subtractions to figure out the volume added (if you filled your burette to 0.00 mL). For each pH reading, simply read off the volume on the burette and that is how much has been added so far.
6. You will notice after a time that your pH readings change by larger increments (> 0.2) - this means you are approaching the equivalence point. When this occurs, begin adding smaller volumes (~0.2 mL) of NaOH solution so that you do not miss the equivalence point.
7. When the pH is 10-12 and adding more NaOH changes the pH by less than 0.3 pH units, your titration is complete.
8. Repeat steps 1-6. Since you now have an idea of where the equivalence point is, you can get higher-quality data the second time. *When titrating around the equivalence point, add NaOH in 0.1 mL increments instead of 1 mL.*

Cleanup

1. Unused NaOH solution can be placed into the collection container.
2. Titration solutions can be discarded down the drain.

Analysis

1. Plot each titration curve with Excel or Sheets - pH (y) vs volume of NaOH added (x). Consult Appendix A for help plotting. Do not fit a trendline.
2. Your plots should have a roughly sigmoidal shape, like the one shown in the background section. Consult your instructor if they do not.
3. Your plots should be printed and taped into your notebook, but you can analyze them on the computer.
4. Identify the equivalence point of each plot. Calculus can be used to precisely identify it, but for our purposes we can determine it visually. Where does the curve seem to change from curving one direction to the other? It will be the most vertical part of the curve.

5. Once you have chosen a point, divide its x value by 2. This x value corresponds to the half-equivalence point. At that x value, trace a line up to the curve. At the intersection point, what is the y value? This is the pK_a.
6. Use Equation 6 to calculate K_a. Do this for both curves. Compare the second value to the acids on the dissociation constant table below to identify the acid.
7. Calculate the concentration of the unknown acid. Do this twice, once for each titration. Start by calculating the moles of base used to reach the equivalence point, using concentration provided and the volume used to reach the equivalence point. Then convert to moles of acid and divide by volume of acid to find the concentration of acid.

Dissociation Constants for Acids at 25°C		
Acid	**Formula**	**Dissociation constant, K_a**
Acetic	$HC_2H_3O_2$	1.8×10^{-5}
Benzoic	$HC_7H_5O_2$	6.5×10^{-5}
Boric	H_3BO_3	5.8×10^{-10}
Chlorous	$HClO_2$	1.1×10^{-2}
Formic	$HCHO_2$	1.8×10^{-4}
hydrocyanic	HCN	4.9×10^{-10}
hydrofluoric	HF	6.8×10^{-4}
Hypobromous	HBrO	2.5×10^{-9}
Hypochlorous	HClO	3.0×10^{-8}
Hypoiodous	HIO	2.3×10^{-11}
Iodic	HIO_3	1.7×10^{-1}
Lactic	$HC_3H_5O_3$	1.4×10^{-4}

6 Buffer Properties

Goals

- Observe how buffers resist changes in pH compared to non-buffered solutions
- Describe the relationship between molar ratio and buffer pH
- Evaluate the effect of buffer concentration and molar ratio on buffer capacity

Pre-lab Questions

1. At what pH is a buffer most effective?
2. Would acetate buffer be a good choice to make a buffer of pH 8?
3. What is the molar ratio of a buffer with $[A^-]$ = 0.15 M and [HA] = 0.50 M?
4. What is the buffer concentration of a buffer with $[A^-]$ = 0.15 M and [HA] = 0.50 M?
5. What is the concentration of sodium acetate in a solution if 4 mL of 0.40 M sodium acetate was added to 46 mL of 0.40 M acetic acid?

- To plot your data, bring a computer to class.

Background

When a solution contains significant amounts of a weak acid and its conjugate base, it acts as a *buffer*. Consider the following equilibrium:

$$HA(aq) + H_2O(l) \rightleftharpoons A^-(aq) + H_3O^+(aq) \quad (1)$$

This buffer solution has been created by mixing equal amounts of the acid and conjugate base. They are in equilibrium - both the forward and reverse reaction are occurring, but the concentration of each component remains constant. If an unrelated strong acid were added to this solution, it would react with the conjugate base (A^-) to form the acid (HA). This disturbs the equilibrium: a certain amount of HA will convert back to A^- according to Le Châtelier's Principle. This keeps the concentration of H_3O^+ relatively consistent, meaning the pH change when the strong acid is added is minimal. The same principle applies if a strong base were added.

This resistance to pH changes isn't limitless; if enough acid or base is added it will *overwhelm* the buffer, fully consuming one of the components of the buffer and significantly changing the pH.

Note that a key requirement for the buffer to function is *significant* amounts of both the acid and conjugate base. Merely adding an acid to water will not create a buffer. Usually the conjugate base is delivered to the solution as a salt. For example, an acetate buffer could be formed by mixing acetic acid with sodium acetate. These components can be added in equal amounts but other ratios are possible.

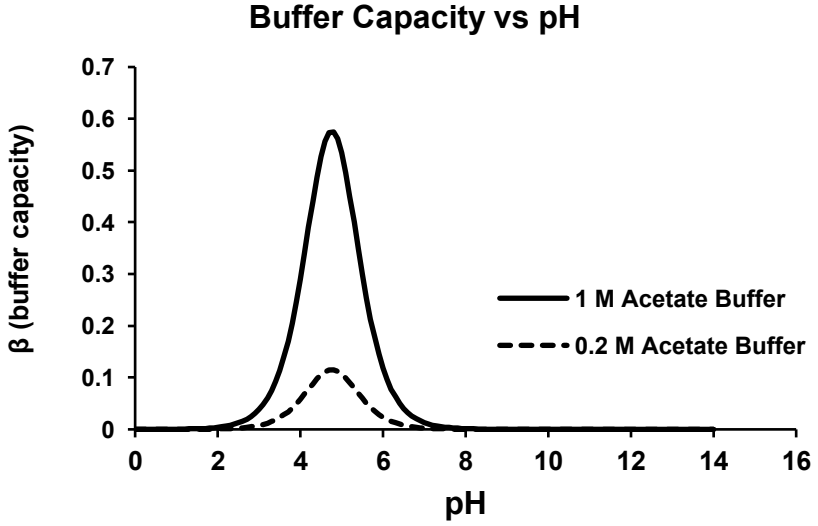
A buffer's *capacity* is a measure of its ability to withstand changes in pH when an acid or base are added to the solution. Buffer capacity (β) is defined as:

$$\beta = \frac{\Delta \text{mol acid or base added}}{\Delta \text{pH}}$$

A higher buffer capacity means it takes more added acid or base to cause a change in pH. Buffer capacity is primarily dependent on two things: the overall *buffer concentration*, and the *molar ratio.* Referring to Equation 1, these factors are defined as:

$[A^-]+[HA]$	$\frac{[A^-]}{[HA]}$
buffer concentration	molar ratio

We can visualize these factors by plotting buffer capacity vs pH of the buffer solution:



**The concentrations refer to total buffer concentration $[A^-]+[HA]$*

The magnitude of buffer capacity decreases proportionally with buffer concentration. This makes sense, as at lower concentrations there is less acid or conjugate base around, and the buffer will be overwhelmed sooner. As the molar ratio changes to favors acid or conjugate base (moving left or right on the x-axis), the buffer capacity also decreases, because having less of the acid or conjugate base component also means the buffer can be overwhelmed sooner.

You may have noticed the buffer capacity curve is centered around the pK_a of the acid (acetic acid $pK_a = 4.75$). A buffer is most effective at a pH equal to its pK_a, and typically ineffective more than 1 pH unit away from the pK_a. This is related to the molar ratio. We will use this experiment to examine this effect.

Procedure (Individual)

Part A: Effect of molar ratio on buffer pH

1. Prepare the five buffer solutions in the table below in separate beakers, and label them.
2. Record the pH of each solution. *Make sure to rinse the probe with distilled water between measurements.*
3. Discard buffers B, D, and E down the drain.
4. Use buffers A and C for the next part.

Buffer	A	B	C	D	E
Volume of 0.5 M NaOAc (mL)	3	8	12.5	17	22
Volume of 0.5 M HOAc (mL)	22	17	12.5	8	3

Part B: Effect of concentration and molar ratio on buffer capacity

1. Prepare 4 beakers and label them:
   - Buffer A (reused from Part A)
   - Buffer C (reused from Part A)
   - Diluted Buffer C: 24 mL of water + 1 mL of Buffer C. Stir to mix.
   - Distilled water, 25 mL
2. Record the pH of each beaker.
3. Add 1 mL of 1 M HCl to each beaker.
4. Stir each beaker with a glass rod to mix in the acid. Rinse the glass rod with distilled water between beakers.
5. Record the pH of each beaker.
6. Mix the contents of all the beakers and pour them down the drain.
7. Remake buffers A, C, and diluted C (and get new distilled water).
8. Repeat steps 1-6 with 1 M NaOH instead of HCl.

Analysis

Part A

1. Calculate the molar ratio for each buffer. This is the ratio between concentration of conjugate base and acid, or $[A^-]/[HA]$. Since the stock solutions of these two components were the same concentration, $[A^-]/[HA]$ = volume A^-/volume HA.
2. To observe how molar ratio influences the pH of a buffer, plot pH vs the molar ratio for each buffer.
3. A linear relationship between two properties is easy to analyze. The relationship between these two properties does not appear to be linear. You

may recognize this curve as a logarithmic function. If we take the log we can get a linear relationship.

4. Calculate the log of each molar ratio, and plot pH vs log($[A^-]/[HA]$). You can use Excel or Sheets to do this by typing "=log(" into a cell and clicking on the cell you want to calculate the log of, and pressing enter.
5. The plot of pH vs log($[A^-]/[HA]$) should be linear. Fit a trendline. The formula for the line is in the format y = mx + b. Using the equation for your line, rewrite the equation using the terms pH and log($[A^-]/[HA]$).
6. How does the y-intercept (b in the formula) of your plot compare to the pKa of acetic acid?
7. Does your equation look similar to anything you've seen before? Maybe in the last experiment?

Part B

Complete this work in your notebook, although you may wish to use the table from Report Question 6 to organize your data.

1. Calculate the molar ratio for each of the three buffers in Part B ($[A^-]/[HA]$).
2. Calculate the total buffer concentration for each buffer ($[A^-]+[HA]$). Use $C_1V_1 = C_2V_2$ to calculate each concentration. You will have to use this equation for both the acid and conjugate base concentrations, then add them to obtain the total buffer concentration. The table of volumes in Part A contains all the information needed.
3. Calculate the change in pH (ΔpH) after reach addition of HCl or NaOH.

$$\Delta pH = |pH_{\text{before addition}} - pH_{\text{after addition}}|$$

4. What do you notice about how well each buffer withstood changes to pH when acid or base was added? How does this compare to pure water?

7 Oxidation-Reduction and Electrochemistry

Goals

- Describe reduction potentials and predict the spontaneity of redox reactions
- Construct a voltaic cell and describe how it works
- Predict and observe the effect of ion concentration on redox reactions

Pre-lab Questions

1. Familiarize yourself with the standard reduction potential table below. What is the reduction potential of Cr^{3+}?
2. Using the standard reduction potentials table in your manual, determine the E°_{cell} for this redox reaction: $3Zn(s) + 2Cr^{3+} \rightarrow 3Zn^{2+} + 2Cr(s)$
3. Is the reaction in number 2 spontaneous? Will it occur?
4. Using Equation 4, determine E_{meas} for the redox reaction $Zn(s) + Cu^{2+} \rightarrow Zn^{2+} + Cu(s)$, if $[Zn^{2+}] = 10$ M and $[Cu^{2+}] = 0.001$ M. You can find E°_{cell} for this reaction below.

Background

Reactions that involve the loss and gain of electrons are known as oxidation-reduction ("*redox*") reactions. This process may be thought of as a combination of two **individual half reactions**, one a reduction, the gain of electrons and the other an oxidation, the loss of electrons.

Consider the reaction that occurs when solid zinc is placed in contact with a solution of copper(II) ions.

$$Cu^{2+} + Zn(s) \rightarrow Cu(s) + Zn^{2+} \qquad (1)$$

The copper ions (Cu^{+2}) are reduced to copper metal ($Cu(s)$). At the same time, zinc metal is oxidized to Zn^{2+}, which goes into solution. We can split this reaction into two half reactions:

$$Cu^{2+} + 2e^- \rightarrow Cu(s) \quad \text{(reduction)} \qquad (2)$$

$$Zn(s) \rightarrow Zn^{2+} + 2e^- \quad \text{(oxidation)} \qquad (3)$$

We can predict when reactions like this are likely to occur. The tendency of a chemical species to acquire electrons (and thereby be reduced) is called its *reduction potential*. The reduction potential, or E°, for any given reaction is measurable by experiment - it is an electrical potential, measured in volts (V).

Electrical potentials are relative values. They can only be measured in comparison to something else. The reduction potential of a proton was arbitrarily assigned a reduction potential of zero and serves as a standard against which other reduction potentials are compared. The values obtained from this comparison are *standard reduction potentials*, and you can find a list of them in the table below.

Standard Reduction Potentials at 25 °C	
Half-reaction	***E°* (V)**
$Ag^{+}(aq) + e^{-} \rightarrow Ag(s)$	+0.799
$Al^{3+}(aq) + 3e^{-} \rightarrow Al(s)$	-1.66
$Ba^{2+}(aq) + 2e^{-} \rightarrow Ba(s)$	-2.90
$Ca^{2+}(aq) + 2e^{-} \rightarrow Ca(s)$	-2.87
$Cd^{2+}(aq) + 2e^{-} \rightarrow Cd(s)$	-0.403
$Co^{2+}(aq) + 2e^{-} \rightarrow Co(s)$	-0.277
$Cr^{3+}(aq) + 3e^{-} \rightarrow Cr(s)$	-0.74
$Cu^{2+}(aq) + 2e^{-} \rightarrow Cu(s)$	+0.337
$Fe^{2+}(aq) + 2e^{-} \rightarrow Fe(s)$	-0.440
$Fe^{3+}(aq) + e^{-} \rightarrow Fe^{2+}(aq)$	+0.771
$2H^{+}(aq) + 2e^{-} \rightarrow H_2(g)$	0.000
$K^{+}(aq) + e^{-} \rightarrow K(s)$	-2.925
$Li^{+}(aq) + e^{-} \rightarrow Li(s)$	-3.05
$Mg^{2+}(aq) + 2e^{-} \rightarrow Mg(s)$	-2.37
$Mn^{2+}(aq) + 2e^{-} \rightarrow Mn(s)$	-1.18
$Na^{+}(aq) + e^{-} \rightarrow Na(s)$	-2.71
$Ni^{2+}(aq) + 2e^{-} \rightarrow Ni(s)$	-0.28
$Pb^{2+}(aq) + 2e^{-} \rightarrow Pb(s)$	-0.126
$Sn^{2+}(aq) + 2e^{-} \rightarrow Sn(s)$	-0.136
$Sn^{4+}(aq) + 2e^{-} \rightarrow Sn^{2+}(aq)$	+0.154
$Zn^{2+}(aq) + 2e^{-} \rightarrow Zn(s)$	-0.763

Using the values in this table, we can calculate an overall potential for a reaction composed of two of the half reactions in the table. This is known as the *standard cell potential*, or E°_{cell} (cell refers to the electrochemical cell these reactions are usually performed in, as seen below.

For redox reactions not taking place in a cell, it may be more accurate to label this term E°_{rxn}). To calculate E°_{cell} we add the standard reduction potentials of our half reactions. However, notice that our half reaction for zinc does not match the one in the table; it is reversed. Before adding these potentials, we must reverse the reaction and thus reverse the sign of the potential, like so:

				***E°* (V)**
$Zn^{2+} + 2e^-$	→	$Zn(s)$	(reduction)	-0.763
$Zn(s)$	→	$Zn^{2+} + 2e^-$	(oxidation)	+0.763

Using that value, we can add the two half reactions:

				***E°* (V)**
$Cu^{2+} + 2e^-$	→	$Cu(s)$	(reduction)	+0.337
	+			
$Zn(s)$	→	$Zn^{2+} + 2e^-$	(oxidation)	+0.763

The E°_{cell} for this reaction is 0.337 V + 0.763 V = +1.100 V.

The E°_{cell} for a spontaneous reaction is positive; the reaction above will spontaneously occur. Conversely, if E°_{cell} is negative, the reaction is not spontaneous.

What about the reverse reaction? Could copper be oxidized and zinc reduced? The E°_{cell} for that reaction would be -1.100 V. This reaction is not spontaneous and not likely to occur.

It is also important that any redox reaction you write is balanced; the same number of electrons should appear on both sides of the equation. This may require multiplying the chemical species by a coefficient. However, this does not affect the standard reduction potential:

			***E°* (V)**
$Fe^{3+} + e^-$	→	Fe^{2+}	+0.771 V
$2Fe^{3+} + 2e^-$	→	$2Fe^{2+}$	+0.771 V

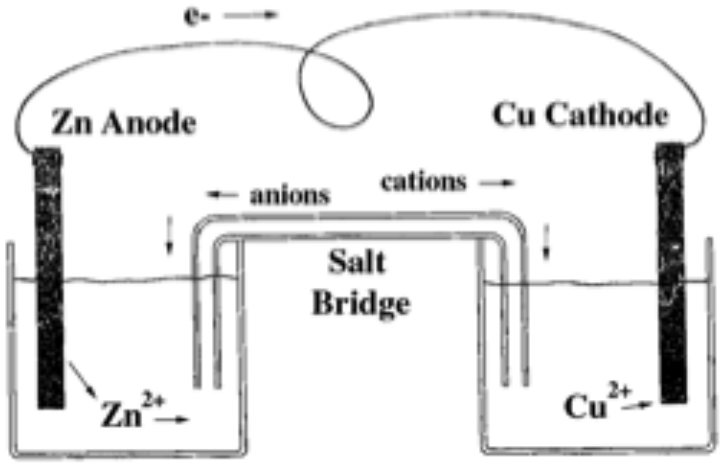
The standard reduction potentials (E^o) of the half-reactions were measured when the concentrations of reactants and products were 1.0 M. If the concentrations are other than 1.0 M, what we observe may differ from the standard conditions. The Nernst equation describes the dependence of the *measured cell potential*, E_{meas}, on the concentrations of the species involved in the reaction. For the Zn-Cu cell, the Nernst equation can be expressed as:

$$E_{meas} = E^\circ_{cell} - \frac{0.0592\ V}{n} \log\left(\frac{[Zn^{2+}]}{[Cu^{2+}]}\right) \qquad (4)$$

In this equation n is the number of electrons exchanged in the redox reaction. Within the logarithm, the concentration of the reduced ion is in the denominator and the concentration of the ion corresponding to the oxidized metal is in the numerator (or products over reactants, considering only the ionic species).

Moving electrons

Electrochemical reactions are typically conducted in an electrochemical cell. The cell is composed of two vessels, each filled with a solution of ions. Into each vessel a piece of solid metal called an electrode is inserted. Ions in solution are free to interact with the electrode and transfer electrons to it. The electrode at which the reduction half reaction takes place is called the cathode, and the electrode where the oxidation half reaction occurs is called the anode. A wire connects these two electrodes, and electrons flow through it in order to complete the reactions. This flow of electrons can be harnessed to do electrical work i.e. run electronic devices like a lightbulb or phone. When a cell functions this way it is called a *voltaic* or *galvanic* cell. The drawing of a cell below shows the copper and zinc reaction described above:



In this image we can see that solid zinc loses electrons, expelling a Zn^{2+} ion into solution. Those electrons travel through the wire connecting the electrodes into the solid copper. Cu^{2+} ions in solution pick up those electrons to become solid copper. As this process occurs charge builds up in each half of the cell - many Zn^{2+} ions are being produced on the left, while a disappearance of the Cu^{2+} ions on the right means the anion that delivered the Cu^{2+} is building up an overall negative charge on the right. To counter this, the salt bridge is added. This is a channel of some background electrolyte, a salt not involved in either half reaction. The salt's component ions are free to move between each half of the cell to balance the charge.

This experiment has two parts. In the first, we will perform redox reactions within test tubes and observe if the standard reduction potentials accurately predict what happens. In the second, we will construct a Zn-Cu voltaic cell and examine the effect of the ion concentrations on the cell potential.

Procedure (Pairs)

Part A: Redox reactions of zinc and copper - predicting spontaneity

1. Fill and label four test tubes to a depth of about 1 cm with the following solutions (each tube receives one solution):
    a. 6 M sulfuric acid
    b. 0.1 M silver nitrate
    c. 0.1 M copper(II) nitrate
    d. 0.1 M sodium nitrate
2. Place a small piece of zinc metal into each of the four tubes.
3. Fill and label four more test tubes to a depth of about 1 cm with the following solutions (each tube receives one solution):
    a. 6 M sulfuric acid
    b. 0.1 M silver nitrate
    c. 0.1 M zinc nitrate (note this is different from the first list)
    d. 0.1 M sodium nitrate
4. Add a small piece of copper metal into each of the four tubes.
5. While the tubes sit, use the standard reduction potentials table to write the redox reaction for each tube. *Hint: The solid metal of each reaction will appear on the reactant side of the overall reaction.*
6. Calculate the overall reduction potential, E°_{rxn} ("E°_{cell}"), for each one. Note whether each reaction is expected to happen.
7. After 10-20 min, examine the solutions for evidence of a reaction and note your observations.

Part B: Zn-Cu voltaic cell - concentration effects on E_{meas}

1. Obtain 75 mL of 0.1 M copper sulfate solution in a 150-mL beaker.
2. Obtain 75 mL of 0.1 M zinc sulfate solution in another 150-mL beaker.
3. Soak a strip of filter paper with saturated KCl solution. This will serve as the salt bridge. Drape the filter paper over the edge of both beakers so that it is touching both solutions.
4. Place a copper electrode in the $CuSO_4$ solution and a zinc electrode in the $ZnSO_4$ solution.
5. Connect the electrodes to a voltmeter and measure the potential of the cell in volts. Turn the dial to the DC voltage area ($V\overline{\overline{\ }}$), and set it do a magnitude of 2 V. Be sure to create a sketch of the reaction setup in your notebook.
6. Remove the copper electrode and add 10 mL of 9 M NH_4OH to the $CuSO_4$ solution. Stir the mixture carefully, noting your observations.
7. Replace the copper electrode. Measure and record the cell potential once it becomes steady.
8. Discard the copper sulfate solution and the salt bridge. Keep the zinc sulfate solution. Rinse the copper sulfate beaker and the copper electrode several times with distilled water and dry both. Fill the beaker as before with fresh 0.1 M copper sulfate solution and prepare a new salt bridge.

9. Reassemble the cell and measure its potential again.
10. Remove the zinc electrode and add 10 mL of 9 M NH_4OH to the $ZnSO_4$ solution. Stir the mixture carefully, noting your observations. The solution should be clear and colorless after all the ammonia is added. If not, add more ammonia in 5 mL increments until it is.
11. Replace the zinc electrode. Measure and record the cell potential once it becomes steady.

Disposal

1. The solutions can be disposed of down the drain.
2. Place intact pieces of copper and zinc metal in the collection jars.
3. Thoroughly rinses the electrodes before storing them.

Analysis

1. If you didn't already, finish writing the reaction equations from Part A and calculating E°_{rxn} for each. An example for sulfuric acid and zinc is shown below:

				***E°* (V)**
	$2H^+ + 2e^-$ →	$H_2(g)$	(reduction)	+0.000
	$Zn(s)$ →	$Zn^{2+} + 2e^-$	(oxidation)	+0.763
+				
	$Zn(s) + 2H^+$ →	$Zn^{2+} + H_2(g)$		+0.763

2. Write the reaction equation for the voltaic cell you constructed and calculate E°_{cell}. Does your initial measured potential (Part B, step 5) agree with what you calculated?

8 Ester Synthesis

Goals

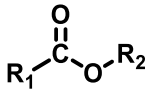
- Describe and draw esters
- Synthesize and purify an ester with reflux, extraction, and distillation
- Determine the product of an esterification based on reagents

Pre-lab Questions

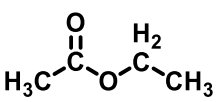
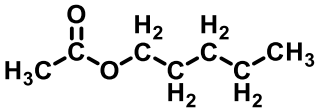
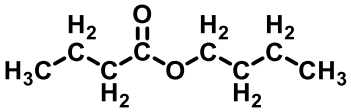
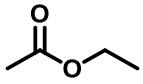
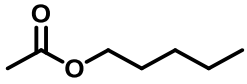
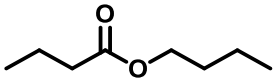
1. What is the limiting reagent in an esterification reaction if 12 mmol of carboxylic acid are used and 11 mmol of alcohol are used?
2. Write the equation for the reaction of sodium bicarbonate with acetic acid.
3. Draw the structure of pentanoic acid and number the carbons it contains.
4. Which carboxylic acid is used to produce ethyl acetate in an esterification?
5. What is the theoretical yield of ethyl acetate if 0.15 moles of acetic acid were used to synthesize it? Assume acetic acid is the limiting reagent.

Background

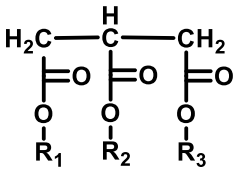
Esters are an important class of organic molecules found in a wide array of substances including nail polish remover, artificial fragrances, fats, oils, and fabric fibers. In chemistry, the term "organic" refers to molecules primarily composed of carbon and hydrogen. Oxygen and nitrogen are also common elements in organic compounds. The basic structure of an ester molecule is shown below.



The "R" groups represent some generic chain of carbons. They do not have to be the same. They may be linear chains or branched, or may include other atoms. What makes it an ester is the presence of the -CO_2- group in between two carbons. Some examples of esters are shown below. From now on, we will display the simplified structure, where the carbons are represented by the corners or ends of lines, and the attached hydrogens are assumed to be present even if not drawn.

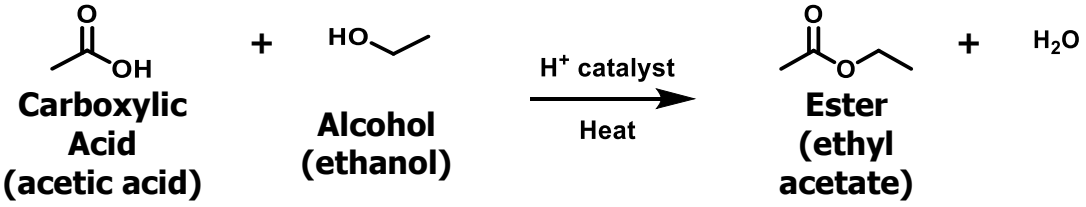
Ester name	Ethyl acetate	Pentyl acetate	Butyl butyrate
Structure			
Simplified Structure			
Common Use	Nail polish remover	Banana flavoring	pineapple flavoring

Fats and oils such as margarine and vegetable oils are triesters, as shown below. The R groups on these molecules are long chains of carbon and hydrogen. The nonpolar character of these chains causes triesters to be insoluble in water.

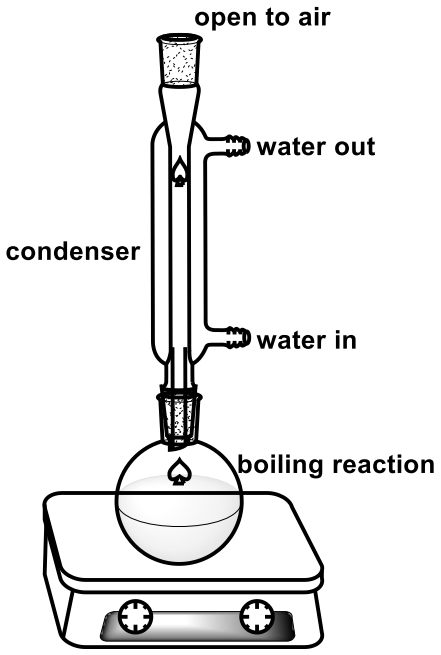


Synthesizing esters

A common way of creating an ester is by combining a carboxylic acid and an alcohol:



This reaction is called an esterification. It produces an ester and a molecule of water. The carbon chain next to the oxygen of the ester comes from the alcohol, and the carbon chain next to the double-bonded carbon comes from the carboxylic acid. This is actually an equilibrium reaction which favors the reactant side of the equation. To promote the formation of the ester, a large excess of either the acid or alcohol is used to push the equilibrium to the right. Adding heat and an acid catalyst such as H_2SO_4 can also promote the reaction.



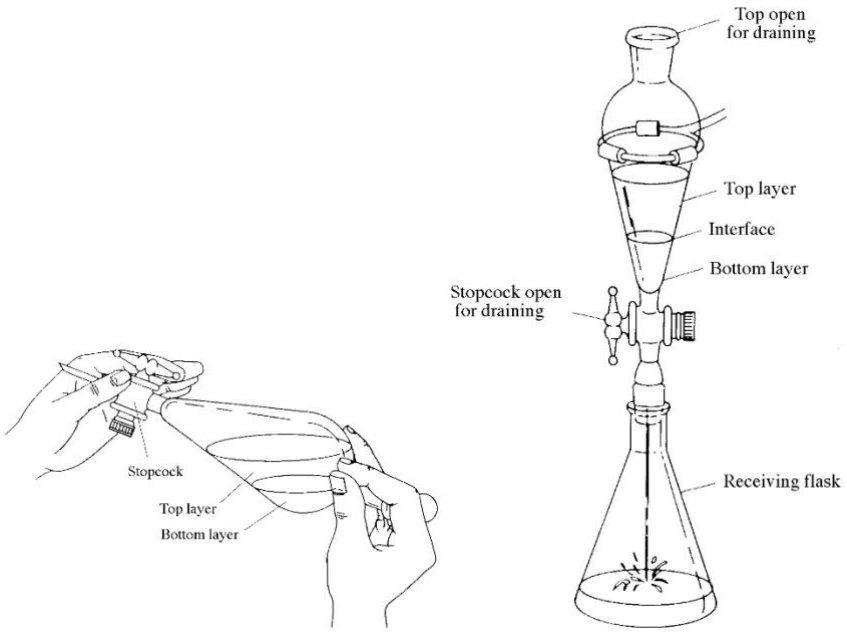
Some special equipment is required to perform this reaction and isolate the ester. We want to add as much heat as possible to drive the reaction forward. Adding a bunch of heat means things are going to start evaporating, thus leaving the reaction flask. To prevent the loss of material, we will conduct a *reflux*. A reflux setup is shown to the right.

The reflux setup is composed of two main features: a heat source, like a hotplate, and a *condenser*. The condenser is a hollow tube within another hollow tube. Inlets allow cool water to flow through the outer, enveloping the inner

tube. As liquids boil, the hot vapor is forced to travel through the condenser, encountering the cold walls of the inner tube and condensing back into liquid. The liquid simply falls back into the reaction flask and the reaction continues without loss of material.

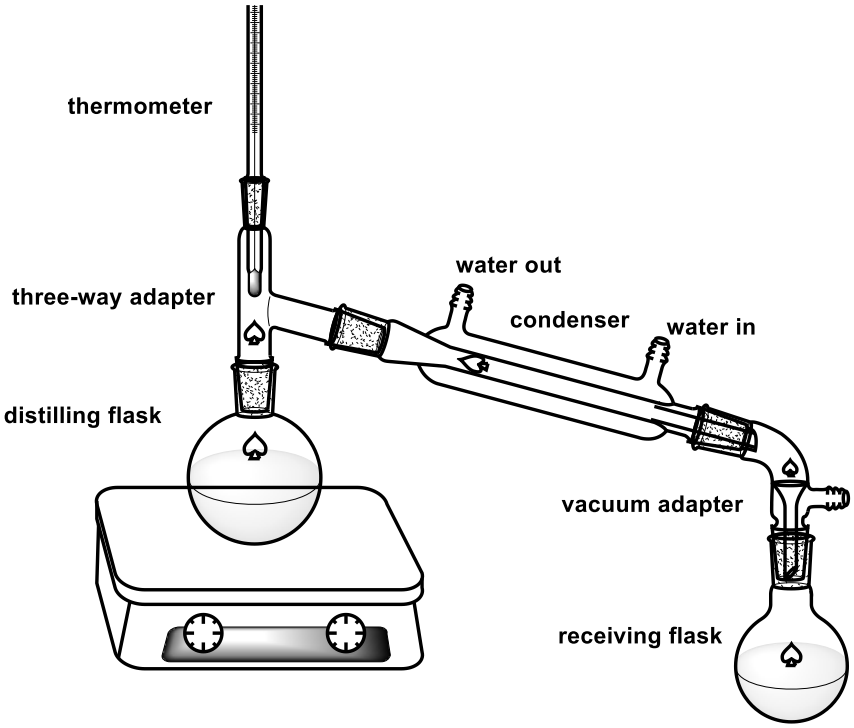
Once the reaction is complete, we have a problem. The ester has formed but there are still plenty of excess reagents leftover. In order to separate our product from the other material, we perform an *extraction*.

Extraction takes advantage of the fact that different molecules have differing solubilities in different solvents. In this case, the ester product is not soluble in water, but the reagents used to make it are! Water can be used to extract the unwanted material out of the reaction mixture. An extraction is performed in a *separatory funnel*, or "sep funnel". The sep funnel has a stopcock on one end and a stopper can be placed in the other end. The reaction mixture and water are poured in the funnel and shaken to ensure thorough mixing. The ester and water will settle as two distinguishable layers, and the unwanted layer can be drained out through the stopcock.



At this point the ester is mostly isolated, but there are still trace impurities leftover. To get a pure product a *distillation* is conducted. Distillation separates substances based on boiling point. If the desired substance has a boiling point sufficiently different from the other components of the mixture, it can be purified with distillation.

The setup for distillation includes a several special pieces of glassware, including a condenser, a three-way adapter, and a vacuum adapter. The material to be distilled is placed in a distillation flask, and distilled material is collected in a receiving flask.



As in the reflux, when liquid evaporates the vapor hits the cold condenser and condenses into liquid, this time flowing into a receiving flask. A thermometer is placed just before the condenser to monitor the temperature of the vapor entering it - the temperature of the vapor at any given point should correspond to the boiling point of the material being distilled. The bulb of the thermometer should be right at the level of the three-way adapter arm that leads to the condenser.

All three of these special pieces of equipment require specific setup steps and careful handling. Follow the directions in the procedure and follow your instructor's instructions for using this equipment.

You will synthesize an ester using a carboxylic acid and an alcohol. For now the identities of these reagents will remain unknown to you. With the analytical data from the next experiment, you will be able to identify your ester and thus the acid and alcohol used to produce it.

Procedure (Pairs)

All work should be conducted within a fume hood. Gloves should be worn at all times.
Be sure to include sketches of the glassware you use for each part of the experiment.

Part A: Synthesis with reflux

1. Using the table below, add the alcohol and acid starting materials to a 100-mL round-bottom flask.
2. Add 1 mL of concentrated H_2SO_4 to the solution with gentle swirling.
3. Assemble a reflux apparatus. Consult your instructor or the example setup in your classroom. Take care to clamp the glassware to a ringstand. Secure each ground-glass connection with a plastic clip.
4. Use rubber tubing to connect the faucet to the lower inlet on the condenser. Attach another piece of tubing from the upper inlet leading toward the drain.
5. Place the flask in a heating mantle.
6. Check with your instructor that your apparatus is proper before continuing.
7. Slowly turn on the water and adjust for a slow but steady flow. The water should be just trickling into the drain.
8. Turn on the heating mantle. The dial should be on the lowest setting that produces vigorous boiling in the flask.
9. Allow the reaction to reflux for 20-25 minutes.
10. Turn off the heating mantle and once cooled, move the flask to a round-bottom stand.

Unknown Ester	Volume of Alcohol (mL)	Volume of Acid (mL)
1	**27.2**	**6.9**
2	**19.0**	**6.9**
3	**25.9**	**6.9**
4	**18.8**	**6.9**
5	**14.6**	**13.0**
6	**14.6**	**9.0**

Part B: Extraction of ester product

1. Put a ring clamp on a ring stand and ensure your sep funnel is held by it. Place a beaker underneath.
2. Close the stopcock of the sep funnel and pour the reaction mixture in.
3. Add ~20 mL distilled water to the sep funnel.
4. Place the stopper in the top of the sep funnel, and hold it in place with one hand while you pick up the funnel. Place another hand on the stopcock, ensuring it stays closed. Consult the illustration in the background section.

5. With the opening of the sep funnel pointing into a fume hood and away from people, shake the sep funnel so the two layers are thoroughly mixed.
6. Upon shaking pressure will build up in the funnel. To vent the pressure, point the opening of the sep funnel into a fume hood, invert the funnel so the stopcock is pointed upwards, and open the stopcock. Close the stopcock and place the funnel back in the ring stand.
7. Remove the stopper and allow the layers to separate. If the layers are having trouble separating, an emulsion may have formed. If this happens add some sodium chloride and cap, shake, and vent the funnel again.
8. Once the layers have separated (and the stopper is removed), open the stopcock to drain the aqueous layer into a beaker. Be sure to close the stopcock before the upper layer drains too.
9. Repeat steps 3-8 but instead of distilled water use 20 mL of sodium bicarbonate solution. The bicarbonate will ensure all residual acid is deprotonated and extracted into the aqueous layer. *The bicarbonate will build up significant pressure, make sure to hold the stopper securely and vent the sep funnel into the hood. This is basically a baking soda volcano.*
10. Repeat steps 3-8 with 20 mL of saturated sodium chloride solution.
11. At this point just the ester layer should be left in the sep funnel. Drain it into a different beaker.
12. Add anhydrous magnesium sulfate. Start with the tip of a spatula. If it clumps up, add some more. Keep adding until there are small, free-flowing particles of magnesium sulfate. This means the $MgSO_4$ has absorbed all the residual water from your ester layer. This is a hydration:

$$MgSO_4(s) + 7H_2O(l) \rightarrow MgSO_4 \cdot \bullet\ 7H_2O(s)$$

13. Decant the ester layer into a clean, dry round bottom flask.

Part C: Purification of ester through distillation

1. Assemble a distillation apparatus. Consult your instructor or the example setup in your classroom. Take care to clamp the glassware to a ringstand. Secure the ground-glass connections with plastic clips.
2. Use rubber tubing to connect the faucet to the lower inlet on the condenser. Attach another piece of tubing from the upper inlet leading toward the drain.
3. Place the flask in a heating mantle and connect it to the apparatus.
4. Check with your instructor that your apparatus is proper before continuing.
5. Weigh a glass vial. This will be used instead of a receiving flask.
6. Slowly turn on the water and adjust for a slow but steady flow. The water should be just trickling into the drain.
7. Turn on the heating mantle. The dial should be on the lowest setting that produces vigorous boiling in the flask.

8. The first few drops of liquid that come out of the condenser ("distillate") may contain impurities - collect these separately and discard. Collect the rest of the distillate in your glass vial.
9. When the flow of distillate is nearly stopped, turn the heat off. Allow the apparatus to cool.
10. Weigh the collection vial to determine how much ester was collected. Label the vial with your name, the unknown number, and the observed boiling point.
11. Waft the ester and describe how it smells.

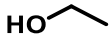
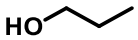
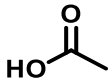
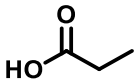
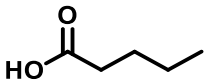
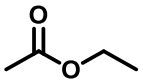
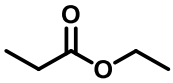
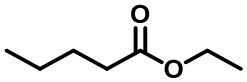
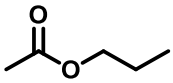
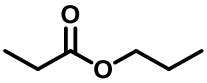
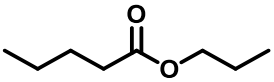
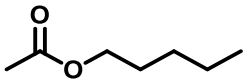
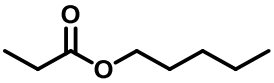
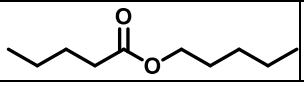
Cleanup

1. Rinse out the glassware with acetone in the hood. Collect the rinse and deposit it in the organic waste disposal container provided.
2. The aqueous layers from the extractions should be poured into the aqueous waste container provided.
3. Magnesium sulfate can be washed down the drain.
4. If boiling chips were used, dispose of them in the trash.
5. Store your labeled vial with your ester as directed by your instructor.

Analysis

The analysis for this experiment is mostly contained within the next experiment. Next week, you will identify the ester you synthesized. The report questions for this experiment will prepare you for the next experiment as well as for writing the Ester Summary Report.

The following page contains a table of chemicals and their properties. You will use this table to answer the report questions for this report and the next experiment's report.

Alcohols, Carboxylic Acids, and Esters					
Chemical	**Structure**	**Formula**	**Molecular Weight (g/mol)**	**Density (g/mL)**	**Boiling Point (°C)**
Ethanol		C_2H_6O	46.07	0.79	78
1-Propanol		C_3H_8O	60.10	0.80	97
1-Pentanol		$C_5H_{12}O$	88.15	0.81	138
Acetic acid		$C_2H_4O_2$	60.05	1.05	118
Propionic acid		$C_3H_6O_2$	74.08	0.99	141
Pentanoic acid		$C_5H_{10}O_2$	102.13	0.94	185
Ethyl acetate		$C_4H_8O_2$	88.11	0.90	77
Ethyl propionate		$C_5H_{10}O_2$	102.13	0.88	99
Ethyl pentanoate		$C_7H_{14}O_2$	130.19	0.88	145
Propyl acetate		$C_5H_{10}O_2$	102.13	0.89	102
Propyl propionate		$C_6H_{12}O_2$	116.16	0.83	123
Propyl pentanoate		$C_8H_{16}O_2$	144.21	0.88	169
Pentyl acetate		$C_7H_{14}O_2$	130.19	0.88	149
Pentyl propionate		$C_8H_{16}O_2$	144.21	0.87	168
Pentyl pentanoate		$C_{10}H_{20}O_2$	172.27	0.87	207

1 Qualitative Analysis of Metal Cations

Goals

- Separate a mixture of ions based on solubility properties
- Confirm the identity of ions through characteristic reactions
- Predict precipitations based on solubility of ionic compounds

Pre-lab questions

1. Complete the separation flowchart on page 3. Fill in the blanks to identify the solid formed in each step and the ion*(s)* left in solution.
2. Write the balanced net ionic reaction equations for the reactions that take place in steps 2, 6, and 10 of the procedure.
3. What color does pH or litmus paper turn when the tested solution is acidic? What color if the solution is basic?
4. Based on the solubility table, what could be added to a solution of Mg^{2+} and Ba^{2+} to separate the two cations?

Background

It is sometimes necessary to identify the chemical contents of a solution. Differences in chemical reactivity can often be used as a means of separating and identifying substances in a mixture. For metal cations in aqueous solution, differences in solubility can be used to separate ions. Further characteristic tests may be conducted to identify ions, often by observing a distinct color in response to a test or reagent.

This experiment explores the use of these chemical and physical properties in separating and identifying several cations in aqueous solution. In Part A, a solution containing four known cations will be subjected to this type of qualitative analysis. In Part B, the same procedure will be applied to a mixture of unknown metal cations in order to identify them.

This experiment will utilize mixtures of the following ions:

Silver, Ag^+ Barium, Ba^{2+} Iron(III), Fe^{3+} Lithium, Li^+

To separate these ions, we can exploit their differing solubilities when combined (reacted) with certain anions. As you can see on the table on the next page, several combinations of cations and anions lead to ionic compounds that are insoluble in water; such combinations would precipitate out of solution and could be separated from anything still dissolved.

Solubility of Ionic Compounds in Water

Anion \ Cation	Li^+	Na^+	K^+	Mg^{2+}	Ca^{2+}	Ba^{2+}	Fe^{2+}	Fe^{3+}	Ni^{2+}	Cu^{2+}	Ag^+	Zn^{2+}	Al^{3+}	Pb^{2+}	NH_4^+
Cl^-	S	S	S	S	S	S	S	S	S	S	I	S	S	I	S
Br^-	S	S	S	S	S	S	S	S	S	S	I	S	S	ss	S
I^-	S	S	S	S	S	S	S		S		I		S	I	S
ClO_3^-	S	S	S	S	S	S		S			S		S	S	S
OH^-	S	S	S	I	ss	S	I	I	I	I	ss	I	I	I	S
S^{2-}	S	S	S		ss	ss	I	I	I	I	I	I	I	I	S
SO_3^{2-}	S	S	S	S	I	I					I	I		I	S
SO_4^{2-}	S	S	S	S	ss	I	S	S	S	S	ss	S	S	I	S
CO_3^{2-}	S	S	S	I	I	I	I		I	I	I	I		I	S
NO_2^-	S	S	S	S	S	S			S		I			S	S
NO_3^-	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
PO_4^{3-}	I	S	S	I	I	I	I	I	I	I	I	I	I	I	S
CrO_4^{2-}	S	S	S	S	S	I	I	I	I	I	I	I		I	S
$CH_3CO_2^-$	S	S	S	S	S	S	S	I	S	S	S	S	ss	S	S

S = soluble, I = insoluble, ss = semi-soluble, blank = no data

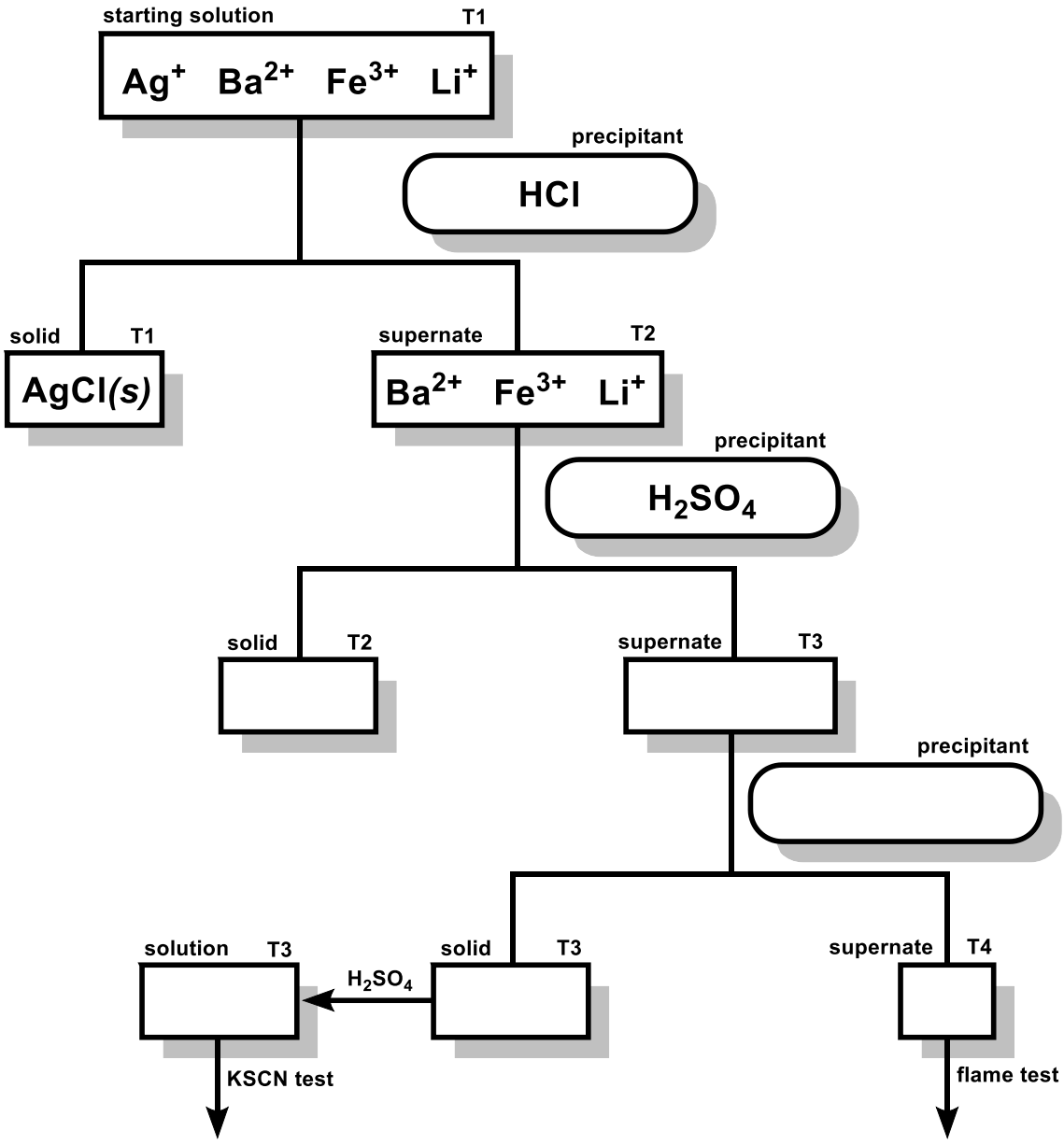
Consider what would happen if we were to add HCl to our mixture of Ag^+, Ba^{2+}, Fe^{3+}, and Li^+. According to the solubility table, Ag^+ would form an insoluble salt with Cl^-.

$$Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s)$$

In this reaction, the added HCl is acting as a *precipitant,* because it causes something to precipitate out of solution. The other ions remain dissolved in the solution because the chloride salts of those ions are soluble. The solid AgCl can be separated by first centrifuging, then decanting away the liquid remaining above the solid, known as the *supernate* or *supernatant*. The supernate contains the remaining three ions that do not form insoluble chlorides. Through this process, the separation of the silver ions has been achieved.

By taking advantage of differing solubilities (as listed in the table), the other ions in the mixture can be separated as well. At each step, one must identify an anion that will form an insoluble compound with only one of the cations remaining in solution. Adding that anion to the solution will allow a solid to precipitate and be separated. Note that semi-soluble combinations should be avoided as they lead to poor separation. If necessary, further reactions can be carried out on the separated materials to identify each ion. If further tests are required, insoluble solids can be dissolved again if converted back into something soluble with an added excess of other reagents.

Separation Flowchart



As mentioned in the pre-lab questions above, complete this flowchart by adding the missing ions, solids, and precipitants. Reading the procedure may help.

Procedure

Be sure to write in your notebook what you are doing and note observations after adding each reagent.

Part A: Separation of stock solution with known cations (Pairs)

1. In a test tube, obtain approximately 3-4 mL of the Ag^+, Ba^{2+}, Fe^{3+}, and Li^+ "Known" solution. Label the tube as Tube 1, or T1.

Separation of first cation

2. Add several drops of 6 M HCl to the solution.
3. Centrifuge the solution to force the solid to the bottom of the test tube. Make sure the centrifuge is balanced - there should always be an even number of tubes, spaced evenly around the carousel. The tubes should contain approximately the same volume/weight of liquid.
4. Add a few more drops of 6 M HCl to the tube. If any cloudiness appears in the solution, precipitation is not complete; repeat steps 2-3. If no cloudiness appears, precipitation is complete.
5. Decant the solution into another test tube, and label it T2.

Separation of second cation

6. Add several drops of 6 M sulfuric acid to T2.
7. Centrifuge the solution.
8. Test for completeness of precipitation with sulfuric acid using an analogous procedure to step 4.
9. Decant the solution into another test tube, and label it T3.

Separation of third cation

10. Add 10-20 drops of 9 M NH_4OH to T3. Use a small spatula to mix.
11. Use pH paper to ensure the solution is basic. If it is not basic, add more 9 M NH_4OH. Red pH paper will turn blue if the solution is basic.
12. Centrifuge the solution.
13. Test for completeness of precipitation with NH_4OH using an analogous procedure to step 4.
14. Decant the solution into another test tube, and label it T4.

Identification of third cation

15. Add 6 M sulfuric acid to the solid remaining in T3 until the solid dissolves. Sulfuric acid introduces H^+ ions which react with the solid in T3 to form water, regenerating Fe^{3+}.
16. To the solution of the dissolved solid, add several drops of 0.1 M potassium thiocyanate, KSCN. Fe^{3+} ions react with thiocyanate in solution to produce a distinctive dark red solution.

Identification of fourth cation

17. Obtain a glass rod with a piece of platinum wire imbedded in it. Light a Bunsen burner and hold the wire in the flame until it burns cleanly and does not change the color of the flame. Be sure to keep the glass rod out of the flame, heating only the wire.
18. Dip the wire into the solution in T4 then hold the wire in the flame. Lithium ions will cause the flame to burn a distinctive ruby red color.

Part B: Identification of cations in an unknown solution (Individual)

1. Obtain an unknown cation solution from your instructor, and note the letter it is labeled with. Each unknown contains some combination of Ag^{+}, Ba^{2+}, Fe^{3+}, and Li^{+}.
2. Repeat steps 2-15 with this solution - note that your unknown may not contain one or more of the cations. Make sure to distinguish this set of test tubes from those used in Part A. If a solid is not formed, you do not need to centrifuge.
3. Using what you know about the solubility of these ions and your observations from the known solution, determine which cations were present in your unknown solution.

Clean-up and disposal

1. Any solids generated need to be collected in labeled waste containers. If you properly label everything, your flowchart should help you identify the solids you generate. A spatula and water wash bottle can be used to get the solids out of the tubes.
2. Remaining solutions (in T3 and T4) can be disposed of down the drain.

Before you leave, make sure to:

- Confirm your separation flowchart is correct
- Confirm you net ionic reaction equation for each precipitation is correct
- Record your unknown number and identify which of the four cations are present in your unknown
- Look over the report questions and ensure you understand them

9 Ester Characterization

Goals

- Identify an ester with the analytical techniques Gas Chromatography-Mass Spectrometry (GC-MS) and Nuclear Magnetic Resonance (NMR) spectroscopy
- Use the identity of the ester to identify the carboxylic acid and alcohol used to synthesize the ester in the previous experiment
- Calculate a yield for the ester synthesize in the previous experiment

Pre-lab Questions

1. On a mass spectrum, what m/z value would the parent peak of propyl propionate have?
2. On a mass spectrum, what would be the m/z value of the base peak of propyl propionate?
3. What would the structure of the fragment corresponding to the base peak of propyl propionate on a mass spectrum look like?
4. How many signals would you expect to see on the carbon NMR spectrum of propyl propionate?
5. Which carbon in propyl propionate would have the highest chemical shift on an NMR spectrum?

Background

Gas Chromatography-Mass Spectrometry (GC-MS) and Nuclear Magnetic Resonance (NMR) spectroscopy are two techniques for characterizing chemical compounds. They are commonly part of the standard characterization done for newly synthesized compounds. This section will give an overview of each technique.

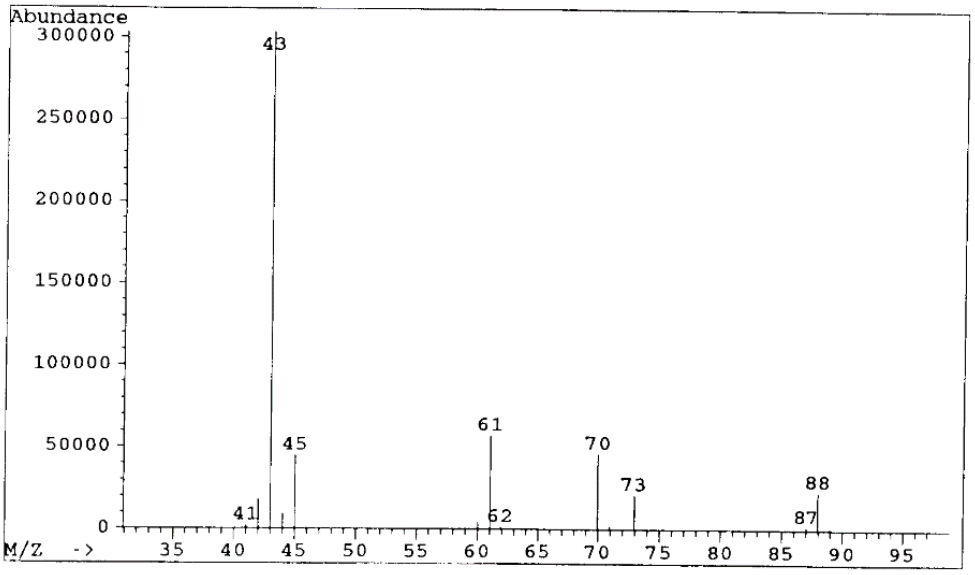
GC-MS

GC-MS is actually composed of two separate techniques - Gas Chromatography and Mass Spectrometry.

Chromatography is a process in which a mixture of chemicals is separated, so that each individual chemical can by analyzed. In Gas Chromatography, a small amount of a liquid sample is injected into a *column*. The column is a tube with some sort of material packed inside, called a *stationary phase.* The stationary phase is some sort of material with a specific property, such as being very nonpolar. Chemicals are carried through the column by the *mobile phase*, which in GC is a gas. As the chemicals travel through the column, some of them stick to the stationary phase

and take longer to come out the other end. In this way, complex mixtures can be separated into individual components. Once this separation takes place, the chemicals will be sent, in the order they come out, to the mass spectrometer portion of the instrument.

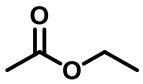
The mass spectrometer can detect the mass of charged particles. It is for this reason that it uses units of mass over charge, or m/z. Since most samples are neutral, a stream of electrons is bombarded at the molecules, causing them to lose an electron and gain a positive charge. The molecular weight of the molecule can then be detected. However, these charged single-electron species are unstable, so they break into *fragments*. The mass of these fragments are also detected, and this is why we see so many peaks on the mass spectrum. Consider the following mass spectrum of ethyl acetate:



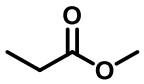
On a mass spectrum there are two important features:

- The *parent peak* is the highest-mass peak on the spectrum. It represents the molecule that has just had a single electron removed, so it has the same mass as the molecule being analyzed. Thus this peak corresponds to the molecular weight of the compound! Here we can see the parent peak of 88 m/z corresponds to ethyl acetate's molecular weight.
- The *base peak* is the tallest peak on the spectrum. A tall peak means many fragments with that mass are present. This peak represents an especially stable fragment. The base peak on this spectrum is 43 m/z.

The mass spectrum can tell us the molecular weight of a compound, but don't discount the fragment peaks! How could we distinguish this spectrum from that of another ester with the same formula and mass as ethyl acetate (a *constitutional isomer*)?

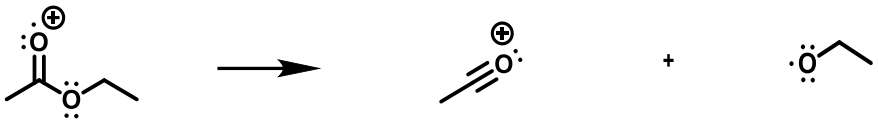


Ethyl acetate
MW = 88.11 g/mol



Methyl propionate
MW = 88.11 g/mol

A common fragment with esters is the one that results when the C-O bond is cleaved:

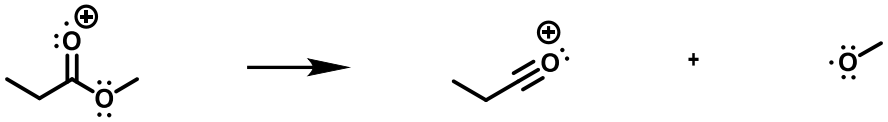


Ethyl acetate
parent ion
m/z = 88

base peak
m/z = 43

Undetected
fragment (no
charge)

This C-O cleavage results in two species: one uncharged fragment including the oxygen and all the carbons on the other side of it, and the carbon with the double-bonded oxygen and all the carbons on that side of the molecule. This second species forms the triple-bonded structure shown, as it is more stable than the double-bonded form. This is called the *oxonium ion*, and if we were to analyze the analogous ion for methyl propionate, we would see it has a m/z value of 57:



Methyl propionate
parent ion
m/z = 88

base peak
m/z = 57

Undetected
fragment (no
charge)

You can calculate m/z values in the same way you calculate molecular weights - just add up the mass of all the atoms. In this way we can determine that our spectrum does in fact correspond to ethyl acetate.

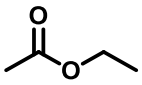
NMR Spectroscopy

NMR spectroscopy is one of the most powerful instrumental methods in chemistry. It is especially useful for identifying organic molecules because it can observe each unique carbon and hydrogen in the molecule. NMR experiments are conducted on one type of nucleus at a time. We will focus on carbon, or ^{13}C-NMR spectroscopy.

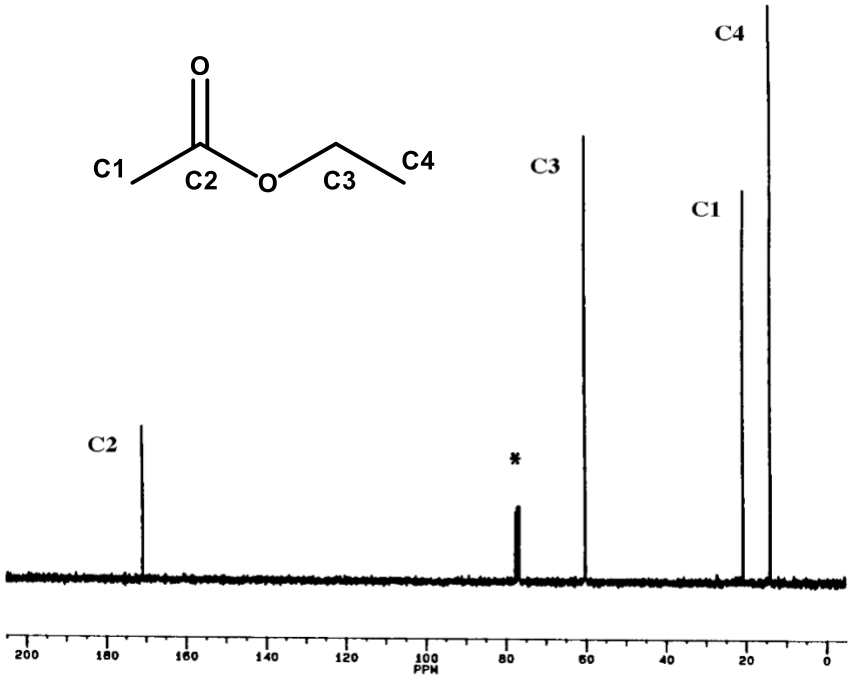
In the NMR instrument, a sample is placed inside a strong magnet. Atomic nuclei align with the magnetic field, and a pulse of light energy is sent into the sample. This perturbs the nuclei out of alignment with the magnetic field. The pulse of light

is stopped and the nuclei gradually realign with the magnetic field, giving off light energy in the process. This light is then measured by the instrument and processed to produce an NMR spectrum.

Not all carbon atoms on a molecule are the same. Ethyl acetate has four carbons:



Each of these carbons is unique, because each of them are attached to different things. The two CH_3 groups on either end are different because one is next to a CH_2 group and the other is next to a C=O group. NMR is so useful because it can distinguish between these unique carbons, allowing us a glimpse at the structure of a molecule. The NMR spectrum for ethyl acetate looks like this:



The x-axis uses units of ppm, and represents the frequency of light each carbon *resonated* at during the experiment. Notice that 0 ppm is on the right side of the plot. The signals are referred to as *chemical shifts*, and each one corresponds to one of the carbons in the molecule.

On this spectrum we can see four peaks, each corresponding to a carbon in ethyl acetate. The peak with an asterisk is due to the solvent the experiment was conducted in, and we will ignore it in our analysis. The chemical shift of a carbon depends on the hybridization and the chemical environment around it:

- *Chemical shifts increase with orbital hybridization in the order $sp^2 > sp > sp^3$*

- *Chemical shifts increase when an electronegative atom is nearby*

Carbon 2 (C2) in ethyl acetate has the largest chemical shift because it is sp^2-hybridized (since it is participating in a double bond) and next to two electronegative oxygen atoms. Likewise, C3 has a somewhat high shift because it is next to an oxygen atom. C1 and C4 have lower chemical shifts, as they are sp^3 hybridized and not connected to any electronegative atoms.

Procedure

You will be provided with an NMR and a mass spectrum of your unknown ester. Follow the steps below to analyze them. You will need to refer to the table of chemicals in the last experiment.

GC-MS Analysis

1. Identify the parent peak in your mass spectrum. Based on this mass, identify which esters from the table this spectrum could represent.
2. Determine the mass or m/z value of the oxonium ion for each of the possible esters identified in the previous step.
3. Identify the base peak on the spectrum. Which oxonium ion does it correspond to?
4. Identify your ester. Which ester did the oxonium ion from the spectrum come from?

NMR Analysis

1. Count the peaks on your NMR spectrum. Each peak represents a carbon in your ester. The exact chemical shifts can be found either in a table below the spectrum or directly next to each peak.
2. Confirm that the number of carbons you counted is consistent with the ester identified with the mass spectrum.
3. Draw your ester on the NMR spectrum. Label the carbons in the ester (C1, C2, etc.).
4. Label the peak that corresponds to the double-bonded carbon bonded to two oxygens.
5. Label the peak that corresponds to the carbon bonded to one oxygen.
6. Label the peaks that correspond to the other carbons. You may not be able to make exact assignments but you can identify a range of peaks that carbon should fall in.

Yield of Synthesis

Now that you've identified your ester, you can evaluate how successful your synthesis was.

1. Identify the carboxylic acid and alcohol that were used to synthesize your ester. Refer to the esterification reaction scheme in the background of the previous experiment.
2. Using the table of chemicals, determine how many moles of each reagent were used. In the esterification, the stoichiometric coefficient of every component is 1. Thus the limiting reagent is whatever reagent you used the fewest moles of.
3. Calculate a theoretical yield of the ester based on the moles of limiting reagent.
4. Calculate a percent yield based on the actual amount of ester you recovered from the distillation.

Ester Summary Report

You must generate a formal scientific report on the synthesis and analysis of your ester. Below are some general considerations for formal scientific writing. The following page includes an outline of what your report should look like. Submit the final report as a PDF file to Moodle.

Your report should:

- Be typed and double spaced, with a standard font and font size.
- Use full sentences
- Be as brief and direct as possible while also including all relevant information
- Use passive voice and past tense
    - "The carboxylic acid was added to the flask" rather than "I added the carboxylic acid to the flask"
- Write this report as if you knew the identity of your ester and reagents from the beginning
- Avoid phrases like "in this experiment". Be direct when describing what was done.
- Avoid referring to "experiment 9" or "experiment 10" etc.
- Spell out acronyms or initialisms before using them
    - Don't write "NMR" without defining it first.
- Do not start a sentence with a number
- Do not capitalize chemical names unless starting a sentence
- Use appropriate superscripts and subscripts for chemical formulas
- Include units with every number
- Include leading zeros in numbers less than one
    - "0.645 g" instead of ".645 g"
- Use ChemDraw structures for chemicals. To download ChemDraw, refer to Appendix F.

Outline of Ester Summary Report

I. Introduction
- This section should give a brief overview of what was made and the evidence that will be used to support that it was in fact made.
- Name the type of reaction that was used to synthesize your product and what the product was
- Name the techniques used to synthesize your product and characterize it, but do not describe the equipment or process yet

II. Synthesis
- This section should allow a chemist of equivalent skill to repeat your synthesis. An example is on the following page.
- Give the names of each reagent, and the amounts of each. You should include the amounts in g or mL and also in moles.
- Walk through the synthesis in order so the reader could repeat it. Mention the reflux, extraction, and distillation as they occur.
- You can assume the reader is familiar with each method. For example, do not describe how to use a separatory funnel, but do state when the extraction took place and what it accomplished.
- Include a ChemDraw reaction scheme for your synthesis with the structures of your reagents and ester. It should look similar to the scheme shown for the ethyl acetate synthesis in the Experiment 9 background.
- State the yield and percent yield you obtained, and describe the product (appearance, aroma, etc.)

III. Results
- This section describes the analysis of your synthesis - how do you know you made what you think you made?
- Describe the most important features of the mass spectrum, the parent and base peaks, and tell how that evidence is consistent with your ester.
- Describe the most important features of the NMR spectrum and tell how that evidence is consistent with your ester.
    i. The number of peaks on the spectrum
    ii. The presence of peaks corresponding to the carbons connected to oxygen. List their chemical shift values with ppm units when referring to them.

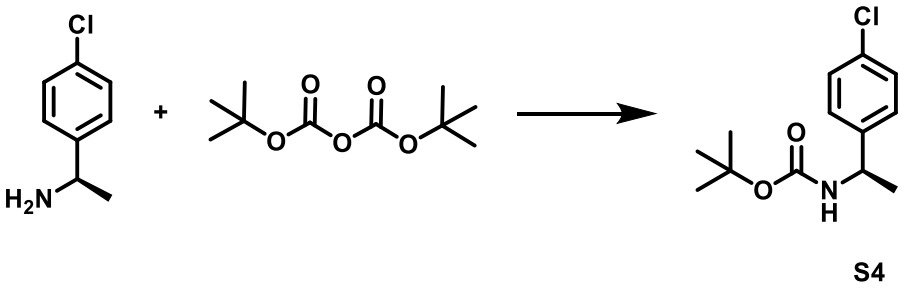
IV. Conclusion
- This section should summarize the supporting evidence that the synthesis was successful. Mention the %yield and the main evidence from NMR and GCMS that confirmed the identity of the product.

Example Synthesis Section

Below is a set of instructions for synthesizing a certain molecule. Your synthesis section should look similar to this. A few things to note:

- The first paragraph describes the reagents and how they were combined - the actual synthesis.
- The second paragraph describes the initial separation, or *workup* of material. Your extraction accomplished a similar task.
- The third paragraph details the purification. This compound was purified with chromatography, while your ester was purified via distillation. The yield and percent yield are stated in this section, along with a description of the product.
- This writeup uses compound codes to refer to chemicals with cumbersome names. Feel free to do this or write out the full name of chemicals you used.

S4, tert-butyl (R)-(1-(4-chlorophenyl)ethyl)carbamate



S4

(R)-(+)-1-(4-Chlorophenyl)ethylamine (126.1 mmol, 19.6 g) and triethylamine (189.1 mmol, 19.1 g, 26.4 mL) were dissolved in dichloromethane (2.72 mL/mmol, 343mL). Di-tert-butyl dicarbonate (Boc_2O, 151.3 mmol, 33 g) was added slowly. The solution was stirred at room temperature for 2 hrs.

The solution was washed with water (2x 200 mL) and brine (1x 200 mL), and dried with $MgSO_4$. After the drying agent was filtered off, the filtrate was concentrated to obtain a white solid.

The white solid was purified with silica gel flash column chromatography (5% ethyl acetate/pentane, Rf = 0.68 in 10% ethyl acetate/pentane) to yield 18.4 g (57% yield) of
aryl chloride **S4** as a white solid.

10 Polymer Synthesis and Properties

Goals

- Understand the structures of polymers and identify the monomeric unit in a polymer
- Synthesize two polymers and study their properties
- Understand the structures of proteins and nucleic acids, important examples of bio-polymers

Pre-lab Questions

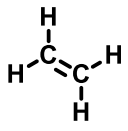
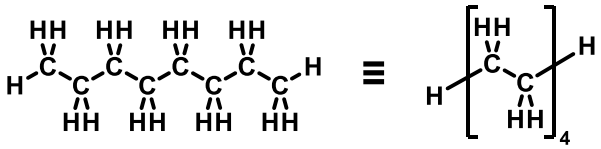
1. What would the condensed structure of polyethylene look like?
2. How many carbons are in the repeat unit for nylon 6,6?
3. How many carbons are in a polyethylene molecule formed from four ethylene monomers?
4. How many carbons are in 1,6-diaminohexane?

Background

Polymers are large molecules composed of many small, similar parts. The Greek prefix *poly* means "many" and the suffix *mer* means "parts". The small part of a polymer is usually referred to as a *monomer* and is chemically linked with other monomers to form a polymer. The result is a very long chain of monomers strung together, sometimes with molecular weights of >1,000,000 g/mol!

Many polymers occur naturally, such as silk, wool, cotton, rubber, proteins, and DNA. Many other polymers are made synthetically, such as plastics like polyethylene or polyester, or synthetic rubber. Many synthetic polymers are known by trade names, such as Styrofoam, Plexiglass, or Teflon. As you can tell from these examples, polymers have a wide variety of functions and uses, from clothing to beverage bottles to car tires to the basic building blocks of life!

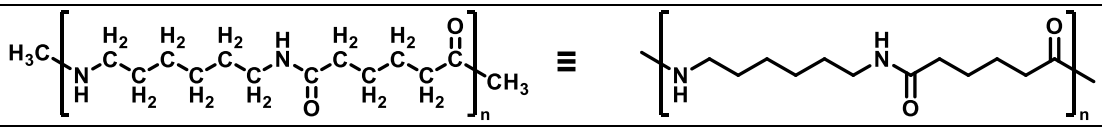
Since polymers can be very long, in order to draw them we can identify the *repeat unit* and place it in brackets, adding a subscript for how many times that piece is repeated in the structure. The end groups will be outside the brackets, as those are only found once. The structure of a short polyethylene is shown below:

	
Ethylene	Polyethylene, expanded and condensed structure

In this experiment, two polymers will be prepared: nylon and a polymer of polyvinyl alcohol known more commonly as "slime".

Nylon 6,10

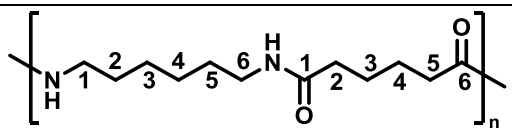
The DuPont chemical company invented nylon in the 1930s. The name "Nylon" originally referred to nylon 6,6, pictured below:



Nylon 6,6

Since there are so many carbons, for clarity we can assume any unlabeled intersection of lines is a carbon, and also assume that each carbon has hydrogens attached such that it has a full octet of electrons, unless otherwise denoted by a labeled formal charge.

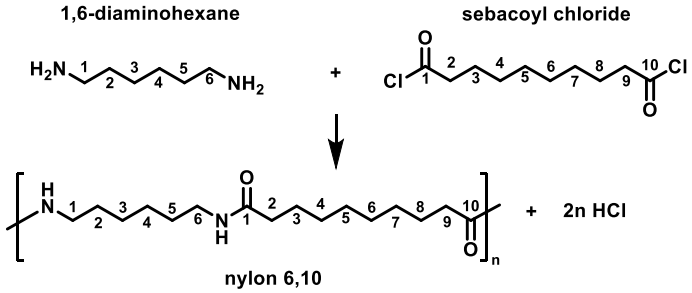
The numbers in nylon 6,6 refer to the number of carbons in between each nitrogen:



Nylon 6,6 with carbons counted

Many other types of nylon were developed with different numbers of carbons so the term "nylon" started to be used to refer to all compounds of this type. These polymers can usually be melted and shaped, and are often extruded into fibers for clothing, string, or rope. Nylon 6,6 was initially used for stockings ("nylons") and quickly found use in parachutes.

In the following synthesis nylon 6,10 is prepared by the *condensation* of sebacoyl chloride and 1,6-diaminohexane. Condensation refers to the fact that a small molecule is lost when two molecules are linked together, in this case HCl:



Synthesis of Nylon 6,10

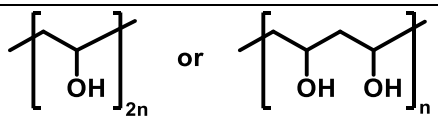
The 6,10 designation is derived from the fact that the polymer chain consists of alternating groups of 6 and 10 consecutive carbon atoms which are separated by the nitrogen atoms. The second number listed includes the carbons double-bonded to oxygen.

This procedure uses an *interfacial polymerization* in which the reagents are separately dissolved in two *immiscible* (non-mixable) solvents. When the solutions are combined, a two-phase system (like oil and water) is produced. The polymer forms as a film at the interface of the two liquid layers.

Cross-linked polyvinyl alcohol ("slime")

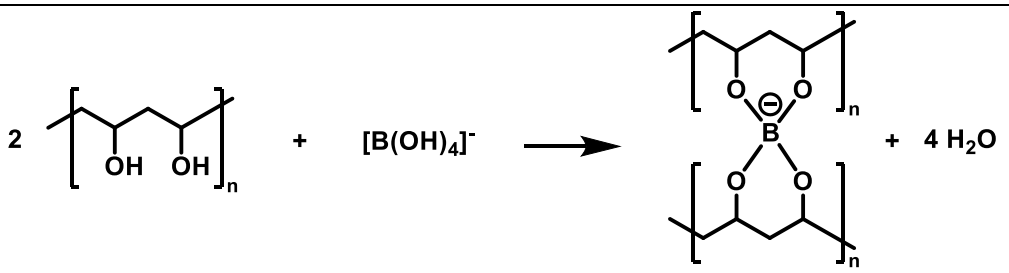
The properties of a polymer can change with *cross-linking.* This means separate strands of polymer are chemically connected to each other to create a new, larger molecule. The physical properties of the material change when cross-linked.

You may have encountered various types of "slime" as a toy or craft. Most of these are some sort of cross-linked polymer. We will make one type of slime today out of poly(vinyl alcohol) (PVA), shown below:



Poly(vinyl alcohol)

Borate ion, **$[B(OH)_4]^-$,** from Borax ($Na_2B_4O_7 \cdot 10H_2O$), is used to cross link the PVA polymer chains into a three-dimensional network of connected chains:



Cross-linking of PVA with Borax

As the polymer is cross-linked water is trapped within it and the material takes on gel-like properties. The degree of cross-linking, and thus the viscosity of the gel, can be varied by changing the amount of borate. From this we can see how the physical properties of a material are dependent on the molecular structure.

Procedure

Part A: Nylon 6,10

1. Add ~5 mL of the 1,6-diaminohexane solution to a small (50-mL) beaker. This solution contains sodium hydroxide which will neutralize the HCl generated.
2. Obtain ~5 mL of sebacoyl chloride solution and gently transfer it to the beaker by slowly pouring down the interior wall, forming a layer that sits on top of the liquid already in the beaker. The two layers should not mix.
3. Allow the reaction mixture to stand undisturbed for about a minute and observe what happens at the interface between the two liquid layers.
4. Using forceps, reach into the center of the beaker and grasp the film that forms at the interface between the two liquids. Slowly raise a polymer strand vertically out of the solution and wrap the top of the strand around the center of a large test tube.
5. Holding the tube at each end, use it as a spool to wind up the strand, taking care to continue drawing the polymer strand from the center of the beaker at a steady rate. If the nylon strand snaps during the spooling procedure, it is possible to generate a fresh strand.
6. Rinse the excess solvent from the surface of the nylon spool by rotating the test tube under a stream of tap water.
7. Slide the nylon from the test tube as an intact loop by inserting a metal spatula between the polymer and the glass surface.
8. Test the tensile strength of the wet nylon by stretching the loop until it snaps.
9. Nylon can be disposed of in the trash after being rinsed.

Part B: Cross-linked polyvinyl alcohol ("slime")

1. Obtain 50 mL of 4% poly(vinyl alcohol) solution in a paper cup. Optional: Add a drop of food coloring to this solution if you want your slime to be colored.
2. Slowly add approximately 6 mL of 4% borax solution. The more you add the stiffer the slime will be, so add just enough to cause polymerization.
3. Stir vigorously with a wooden stick. Once the solid gel has formed, remove it and wash it with water to remove excess base.
4. Test the physical properties of the gel by stretching it, placing it on the bench top, pulling it apart, and reworking it into a single mass.
5. If you wish, you may keep your "slime". Keep it in a plastic bag and be careful not to get in on painted furniture or carpet. It can be disposed of as normal garbage.

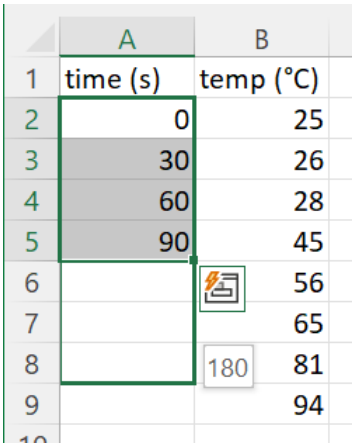
Appendix A: Plotting Data Electronically

It is useful to plot data in a spreadsheet program such as Microsoft Excel or Google Sheets. These programs enable the quick manipulation of data and allow precise graphs or plots to be generated. Rapid calculations can also be performed by entering formulas into cells. Both of the programs mentioned above have an abundance of documentation online; if you want to learn how to do something specific you can often find a tutorial or forum post detailing how it is done by searching the internet.

This appendix will give a brief overview of how to perform the most common functions in each of these programs.

Entering data

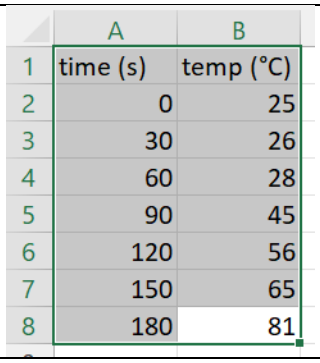
The spreadsheet is a series of rows and columns, labeled with numbers and letters respectively. One should enter all of one type of data in a column, such as all time measurements or all temperature measurements. You may want to add column headers and unit labels so you remember what is what.

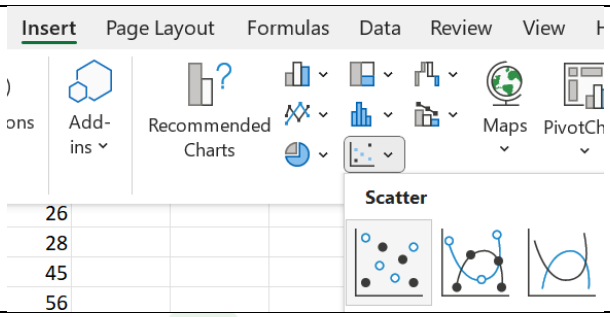
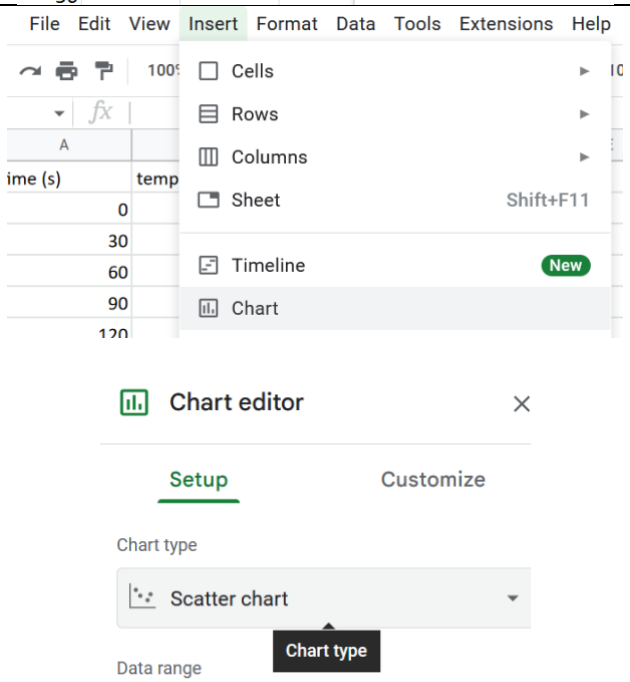
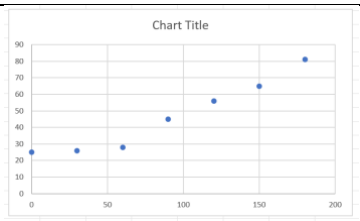
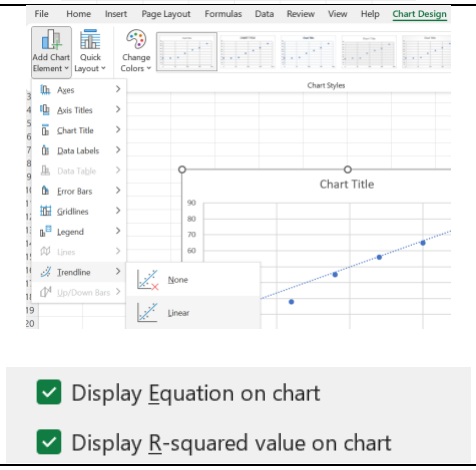


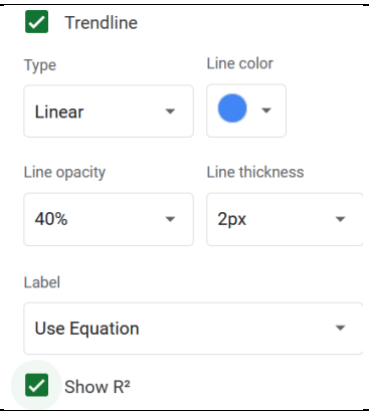
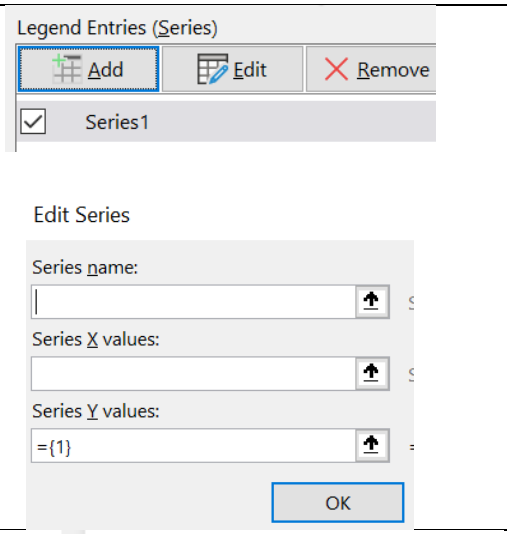
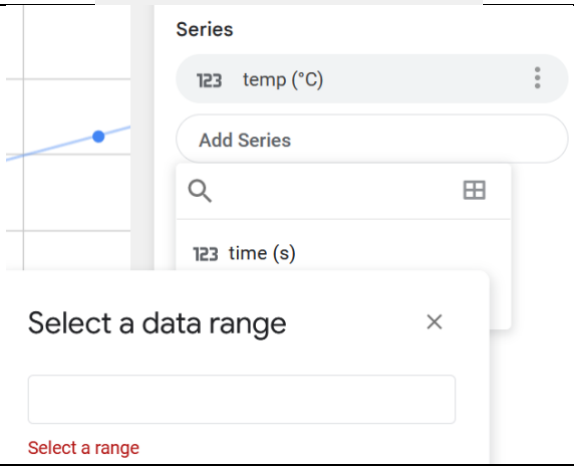
If you are incrementing some values by regular intervals, you can usually enter just the first few, select those cells, and click and hold the small box in the corner and drag down to autofill the other cells.

It is customary to put your x values or independent variable in the first column and y values or dependent variables in following columns.

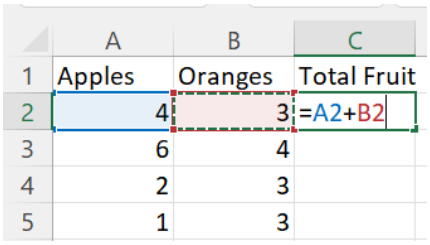
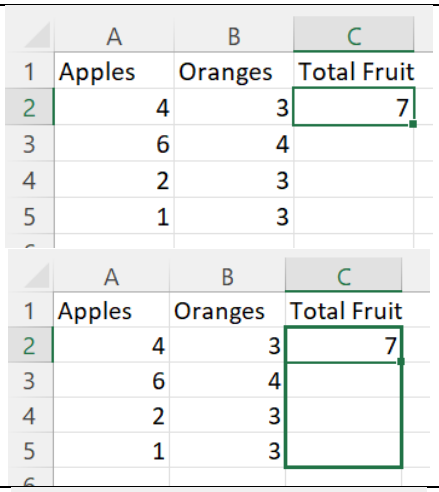
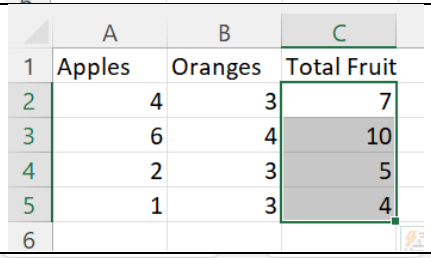
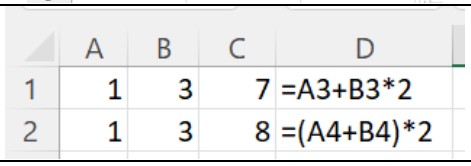
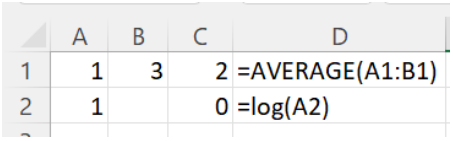
Plotting data

Once you have entered your data, click and drag so it is all selected.	

In Excel, click "Insert" on the ribbon or menu bar and select chart or click the scatter icon and select the normal scatter plot, with no lines between points.	
In Sheets, click "Insert" in the menu bar and select chart. Warning - if you do not have column headers in Google Sheets, the program will mess up. Once the plot is inserted the Chart editor should appear as a sidebar - Under the Setup tab, use the Chart type dropdown to select Scatter chart. If the chart editor does not appear, click the 3 dots in the upper right of the plot and select edit.	
You should now have a basic plot.	
To add a trendline in excel, slect the chart, then in the ribbon click Chart Design>Add Chart Element>Trendline. Typically you want to use Linear, but other curves are sometimes used. Right click on the trendline and select "Format Trendline". The sidebar should open with checkboxes for displaying the equation and R^2 value on the chart. Select both.	

To add a trendline in Sheets, open the Chart editor and under the Series dropdown you will find a checkbox for Trendline. For Type you typically want to use Linear, but other curves are sometimes used. Use the "Label" dropdown to select "Use Equation" and check the "Show R^2" box.	
If you want to plot other data, you can other add data series to your plot.	
In Excel, right-click the plot and select "Select Data". There should be a button to add a series. Clicking that opens up the Edit Series dialog box, and you can click the arrow icons to select the range of data on the spreadsheet you want. The "Series name" is what will appear in the legend of the plot.	
In Sheets, open the Chart editor and under the Setup tab select the Add Series button. Click the table icon and a box appears. You can click and drag to select another data range.	
This second data series can have its own trendline and formatting.	

Performing calculations in a spreadsheet

Both Excel and Sheets allow for calculations to be performed on data via the use of formulas. The basic usage of formulas look the same in both programs.	
Let's look at an example of adding the data in two columns. Start the formula by typing "=" into a cell. Then use any mathematical operators (+, -, *, /). You can type "A1" and "A2" or simply click on the cell you want to refer to.	
Pressing enter will perform the calculation. To perform the same calculation with other rows, click on the cell to highlight it, then click and hold the small box in the corner and drag down to highlight the rest of the column.	
After releasing the click, the formula will transfer to the other cells and automatically calculate.	
The program will follow order of operations unless parentheses are used. Column D shows the formula typed into column C.	
You can also use functions for more advanced operations. Column D shows the formula typed into column C.	

Preparing plots for presentation

Any plot that you put in a report or present to an audience should have at minimum:

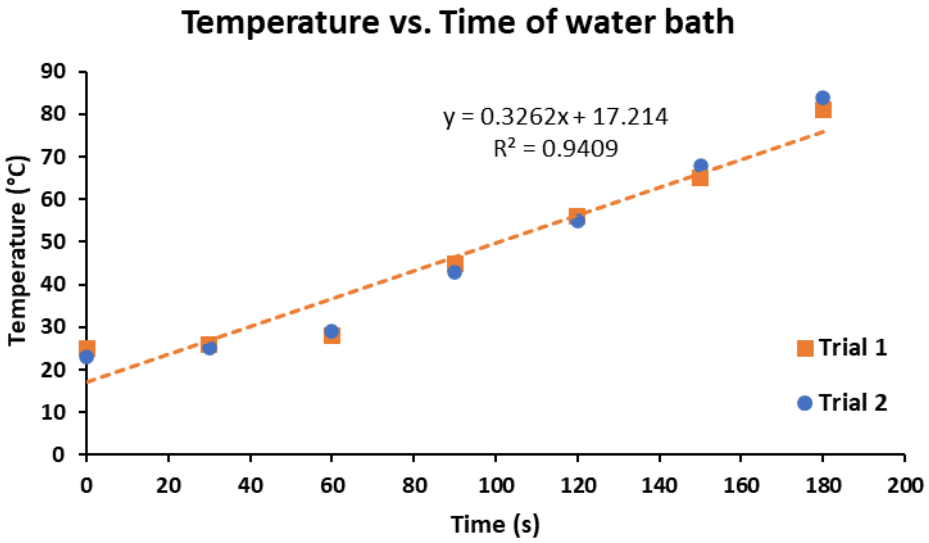
- X- and y-axis labels, including units
- A legend if more than one data series is displayed
- A title (or caption) describing what the plot is showing - this can often just be dependent variable vs. dependent variable or y-axis vs. x-axis

You may also wish to include:

- A trendline for each data series, with the equation and R^2 value displayed
- Formatting to make it visually appealing - colors, different shapes for points, removed lines, bolded axis numbers, etc.

Options for many of these things can be found in the Chart editor Customize tab in Google Sheets or in the Chart Design or Format tabs in the Excel ribbon. Before using the Format tab in excel, click on the individual element in the chart you wish to edit.

When you are ready to share your plot, select the whole thing and copy/paste it into your word processor. A publication-ready plot is shown below:



Appendix B: Sample Notebook Entry

Date 9/16/2021

Title Exp. 3: Gravimetric Analysis of a Chloride Salt

Purpose: Determine % chloride in an unknown an unknown solid. The solid will be dissolved and $AgNO_3$ added to cause precipitation of AgCl solid, which can be filtered and weighed to determine % chloride.

Reaction: $Ag^+_{(aq)} + Cl^-_{(aq)} \rightarrow AgCl_{(s)}$

Net ionic equation is written

Procedure:	Observations:
① Weigh 3 samples of unknown, ~0.25g, label A, B, C	unknown #47, white solid A: 0.2536g, B: 0.2530g, C: 0.2528g.
② Dissolve each in 75mL H_2O	solutions are colorless
③ Add 25mL 0.2 M $AgNO_3$	solutions turned cloudy, white

Page number 12

(4) Solutions gently heated, covered with foil	white solid clumped together heat up to ~40 °C
(5) 3 filter papers labeled A, B, C	A: 0.8637 g, B: 0.8832 g C: ~~0.8784 g~~ 0.8736 g Mistakes are simply crossed out
(6) Solutions gravity filtered through pre-weighed filters	collected solid is grey/white
(7) Filter papers + solid allowed to dry overnight	stored in lab drawer
9/17/2021	
(8) Papers with solid weighed	A: 1.4864 g, B: 1.5049 g C: 1.4934 g

Everything above except for observations can be completed before class

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Data:

mass (g)	A	B	C
unknown solid	0.2536	0.2530	0.2528
empty filter paper	0.8637	0.8832	0.8736
filter paper w/ solid	1.4864	1.5049	1.4934
AgCl solid	0.6227	0.6217	0.6198

You could construct the empty table before class as well

Calculations:

For each (A, B, C) find g Cl^- in AgCl solid and then % of original unknown mass.

A: $0.6227\ g\ AgCl \cdot \frac{1\ mol\ AgCl}{143.32\ g} \cdot \frac{1\ mol\ Cl^-}{1\ mol\ AgCl} \cdot \frac{35.45\ g\ Cl^-}{1\ mol} = 0.1540\ g\ Cl^-$

$\frac{0.1540\ g\ Cl^-}{0.2536\ g\ unknown} \cdot 100 =$ **60.74% Cl^-**

B: $0.6217\ g\ AgCl \cdot \frac{1\ mol\ AgCl}{143.32\ g} \cdot \frac{1\ mol\ Cl^-}{1\ mol\ AgCl} \cdot \frac{35.45\ g\ Cl^-}{1\ mol} = 0.1538\ g\ Cl^-$

$\frac{0.1538\ g\ Cl^-}{0.2530\ g\ unknown} \cdot 100 =$ **60.78% Cl^-**

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C: $0.6198\ g\ AgCl \cdot \frac{1\ mol\ AgCl}{143.32\ g} \cdot \frac{1\ mol\ Cl^-}{1\ mol\ AgCl} \cdot \frac{35.45\ g\ Cl^-}{1\ mol} = 0.1533\ g\ Cl^-$

$\frac{0.1533\ g\ Cl^-}{0.2528\ g\ unknown} \cdot 100 =$ **60.64 % Cl^-**

Average: $\frac{60.74 + 60.78 + 60.64}{3} =$ **60.72% Cl^-**

Conclusion: The unknown solid contained an average of 60.72% chloride.

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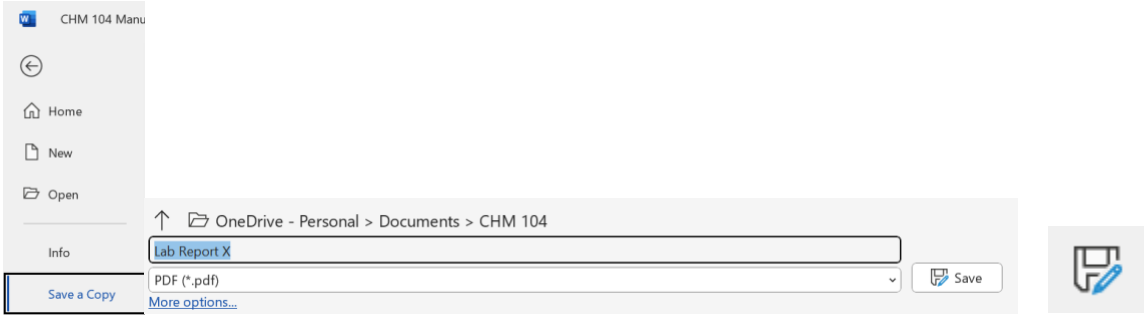
Appendix C: Using PDF files

All reports should be submitted as portable document format (PDF) files. This format not only makes it easier for your audience to view your work, it also preserves any formatting you applied so the document appears identical regardless of device or software used to view it. If you are not familiar with this file format, below are a few ways to save your work as a PDF file.

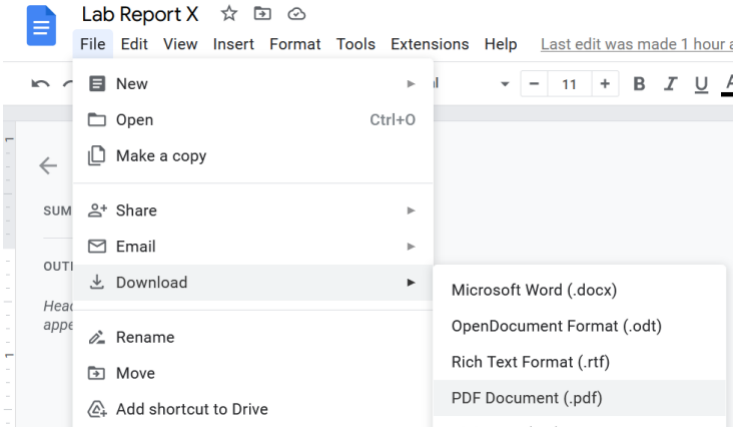
Saving to PDF using Microsoft Word

In Word (or other Office apps), select "File>Save As" or "Save a Copy" and use the dropdown menu to select "PDF".



Saving to PDF using Google Docs

In Docs go to File>Download>PDF Document.



Saving to PDF from handwritten work

Handwritten or physical documents (such as lab notebook pages) can be scanned into a PDF file using most smartphones. Your phone may already have this functionality, or you may need a 3rd party app. You can search for "document scanner" apps to find one.

Alternatively, you can take photos of the documents and put them in an electronic document, then follow the directions above.

Appendix D: Calculating Molar Mass

The molar mass of a chemical tells us how much of it is contained in a certain weight. When performing chemical reactions, it is important to know how many particles of a substance you are reacting. The molar mass allows us to practically measure amounts of a substance to work with.

Molar masses are reported with units of grams per mole (g/mol) and are often used in dimensional analyses.

Often molar masses can simply be looked up, but with a periodic table you can always calculate them by hand. A molecule's molar mass is simply the *sum of the atomic masses of all the atoms in the molecule*, which can be located on many periodic tables (including the one in this manual).

Example

Calculate the molar mass of acetic acid, CH_3COOH (or $C_2H_4O_2$).

There are three elements in acetic acid: carbon, hydrogen, and oxygen. They have an atomic masses of 12.01, 1.008, and 16.00 respectively. There are 2 carbons, 4 hydrogens, and 2 oxygens. Thus the molar mass of acetic acid can be expressed as:

$$\text{molar mass } C_2H_4O_2 = 2(12.01) + 4(1.008) + 2(16.00) = 60.05 \text{ g/mol}$$

The molar mass of acetic acid is 60.05 g/mol.

Appendix E: Uncertainty in Measurement and Significant Figures

Most measurements include some degree of uncertainty. It is important that our reported values indicate how certain our measurements are, so we do not mislead our audience or make erroneous claims about our data. In any number, the digits that we can be reasonably certain of are called **significant figures** (SF).

Recording significant figures

The equipment used to measure a value dictates the number of SF.

- Electronic balances simply display all significant figures - record the number as displayed.
- Volumetric glassware has been calibrated to a certain degree of precision, if used properly. The SF are often listed directly on the glassware (e.g. a volumetric pipette might have 25.00 mL written on it. Record this value exactly, with the trailing zeroes.)
- Equipment with markings interpreted by eye, such as a ruler or graduated cylinder, are read to the nearest digit not indicated by the markings. For example, a ruler may be marked every millimeter, but must be read to the nearest 0.1 mm. A graduated cylinder marked every 10 mL must be read to the nearest 1 mL. The last digit in these cases may need to be estimated.

What counts as a significant figure?

All non-zero digits are always significant. The digit zero may or may not represent a significant figure depending on context.

Significant zeroes include:

- Zeroes in between other digits
  - 1.001 (4 SF), 200.1 (4 SF)
- Zeroes trailing a decimal, if no other digits are present after the decimal
  - 53.00 (4 SF), 50.00 (4 SF)
- Zeroes trailing a decimal, if present after other digits
  - 0.00400 (3 SF), 10.010 (5 SF)

Insignificant zeroes include:

- Leading zeroes
  - 0.0000056 (2 SF), 0.13 (2 SF), 012 (2 SF)
- Trailing zeroes in an integer, unless a decimal point is included
  - 1500 (2 SF), 1500. (4 SF)

Appendix F: ChemDraw

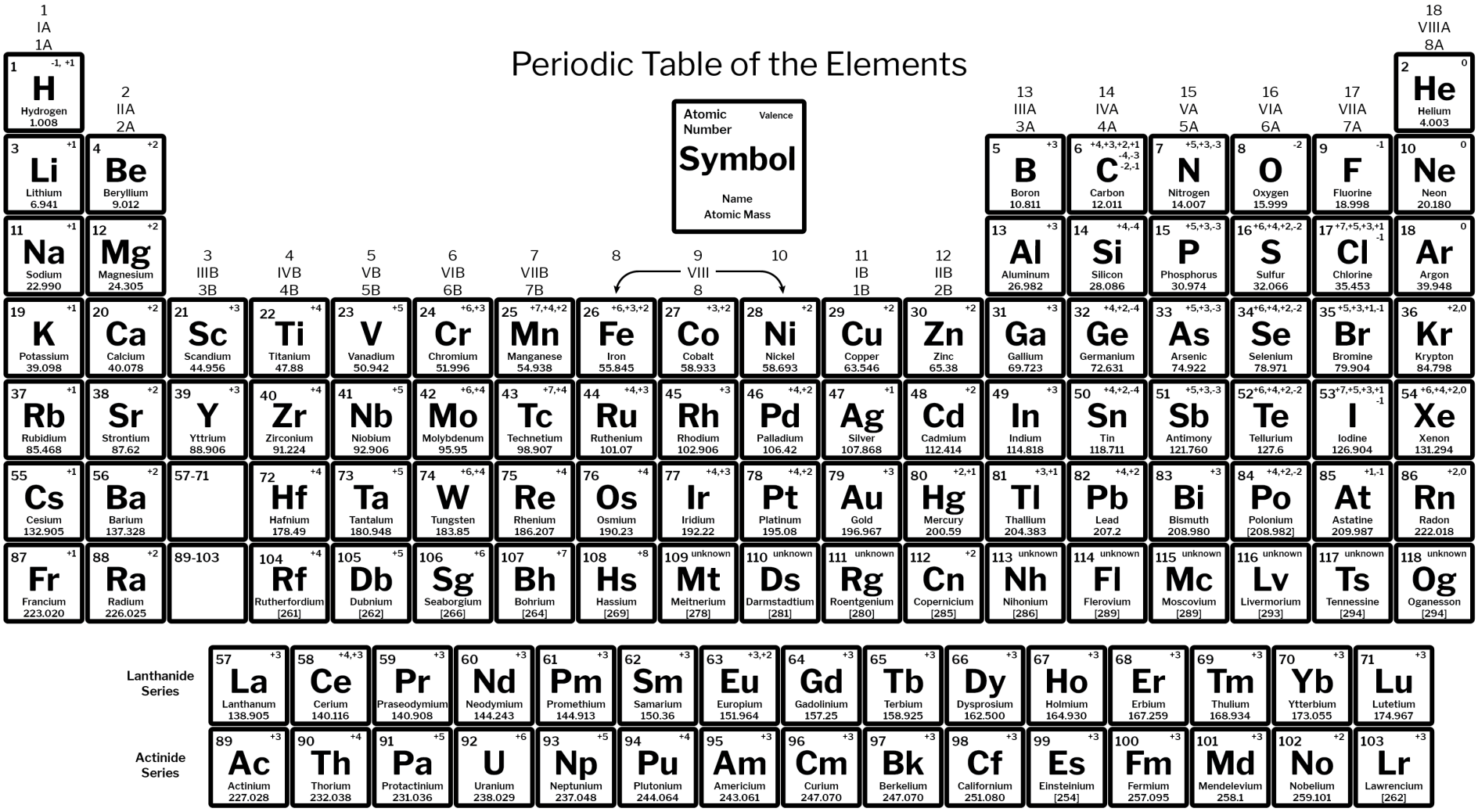
ChemDraw is professional-grade software used to draw chemical structures, especially for organic chemistry. All formal chemistry communication utilizes computer-drawn structures. This software is included in your tuition at Connecticut College, and you will use it often if you take organic chemistry here.

To download ChemDraw:

- Go to https://connect.revvitysignals.com/sitesubscription/
  - Sometimes this URL changes – you can usually find the right page by searching "chemdraw site subscription". The page should have a search field and a list of universities.
- Use the search field to find Connecticut College
- Click the Register button (the top one) and make sure to use your Conn email to register. You can use the college's address if you wish (270 Mohegan Avenue)
- Download the software for Windows or macOS
- Alternatively, some public computers on campus have ChemDraw installed.
- There are numerous tutorials online for how to use ChemDraw. Here is one: https://youtu.be/a9r4Ofnc-Ro?t=297
- You should be able to copy and paste from ChemDraw into your word processor (may not work for Google Docs). Alternatively, you can save the ChemDraw file as an image file like .png and add that to your document.

If you run into issues installing or using ChemDraw, consult your instructor.

Periodic Table of the Elements


1 IA 1A
2 IIA 2A
3 IIIB 3B
4 IVB 4B
5 VB 5B
6 VIB 6B
7 VIIB 7B
8
9 VIII 8
10
11 IB 1B
12 IIB 2B
13 IIIA 3A
14 IVA 4A
15 VA 5A
16 VIA 6A
17 VIIA 7A
18 VIIIA 8A
Atomic Number
Valence
Symbol
Name
Atomic Mass
1 -1, +1 H Hydrogen 1.008
2 0 He Helium 4.003
3 +1 Li Lithium 6.941
4 +2 Be Beryllium 9.012
5 +3 B Boron 10.811
6 +4,+3,+2,+1 -4,-3 -2,-1 C Carbon 12.011
7 +5,+3,-3 N Nitrogen 14.007
8 -2 O Oxygen 15.999
9 -1 F Fluorine 18.998
10 0 Ne Neon 20.180
11 +1 Na Sodium 22.990
12 +2 Mg Magnesium 24.305
13 +3 Al Aluminum 26.982
14 +4,-4 Si Silicon 28.086
15 +5,+3,-3 P Phosphorus 30.974
16 +6,+4,+2,-2 S Sulfur 32.066
17 +7,+5,+3,+1 -1 Cl Chlorine 35.453
18 0 Ar Argon 39.948
19 +1 K Potassium 39.098
20 +2 Ca Calcium 40.078
21 +3 Sc Scandium 44.956
22 +4 Ti Titanium 47.88
23 +5 V Vanadium 50.942
24 +6,+3 Cr Chromium 51.996
25 +7,+4,+2 Mn Manganese 54.938
26 +6,+3,+2 Fe Iron 55.845
27 +3,+2 Co Cobalt 58.933
28 +2 Ni Nickel 58.693
29 +2 Cu Copper 63.546
30 +2 Zn Zinc 65.38
31 +3 Ga Gallium 69.723
32 +4,+2,-4 Ge Germanium 72.631
33 +5,+3,-3 As Arsenic 74.922
34 +6,+4,+2,-2 Se Selenium 78.971
35 +5,+3,+1,-1 Br Bromine 79.904
36 +2,0 Kr Krypton 84.798
37 +1 Rb Rubidium 85.468
38 +2 Sr Strontium 87.62
39 +3 Y Yttrium 88.906
40 +4 Zr Zirconium 91.224
41 +5 Nb Niobium 92.906
42 +6,+4 Mo Molybdenum 95.95
43 +7,+4 Tc Technetium 98.907
44 +4,+3 Ru Ruthenium 101.07
45 +3 Rh Rhodium 102.906
46 +4,+2 Pd Palladium 106.42
47 +1 Ag Silver 107.868
48 +2 Cd Cadmium 112.414
49 +3 In Indium 114.818
50 +4,+2,-4 Sn Tin 118.711
51 +5,+3,-3 Sb Antimony 121.760
52 +6,+4,+2,-2 Te Tellurium 127.6
53 +7,+5,+3,+1 -1 I Iodine 126.904
54 +6,+4,+2,0 Xe Xenon 131.294
55 +1 Cs Cesium 132.905
56 +2 Ba Barium 137.328
57-71
72 +4 Hf Hafnium 178.49
73 +5 Ta Tantalum 180.948
74 +6,+4 W Tungsten 183.85
75 +4 Re Rhenium 186.207
76 +4 Os Osmium 190.23
77 +4,+3 Ir Iridium 192.22
78 +4,+2 Pt Platinum 195.08
79 +3 Au Gold 196.967
80 +2,+1 Hg Mercury 200.59
81 +3,+1 Tl Thallium 204.383
82 +4,+2 Pb Lead 207.2
83 +3 Bi Bismuth 208.980
84 +4,+2,-2 Po Polonium [208.982]
85 +1,-1 At Astatine 209.987
86 +2,0 Rn Radon 222.018
87 +1 Fr Francium 223.020
88 +2 Ra Radium 226.025
89-103
104 +4 Rf Rutherfordium [261]
105 +5 Db Dubnium [262]
106 +6 Sg Seaborgium [266]
107 +7 Bh Bohrium [264]
108 +8 Hs Hassium [269]
109 unknown Mt Meitnerium [278]
110 unknown Ds Darmstadtium [281]
111 unknown Rg Roentgenium [280]
112 +2 Cn Copernicium [285]
113 unknown Nh Nihonium [286]
114 unknown Fl Flerovium [289]
115 unknown Mc Moscovium [289]
116 unknown Lv Livermorium [293]
117 unknown Ts Tennessine [294]
118 unknown Og Oganesson [294]
Lanthanide Series
57 +3 La Lanthanum 138.905
58 +4,+3 Ce Cerium 140.116
59 +3 Pr Praseodymium 140.908
60 +3 Nd Neodymium 144.243
61 +3 Pm Promethium 144.913
62 +3 Sm Samarium 150.36
63 +3,+2 Eu Europium 151.964
64 +3 Gd Gadolinium 157.25
65 +3 Tb Terbium 158.925
66 +3 Dy Dysprosium 162.500
67 +3 Ho Holmium 164.930
68 +3 Er Erbium 167.259
69 +3 Tm Thulium 168.934
70 +3 Yb Ytterbium 173.055
71 +3 Lu Lutetium 174.967
Actinide Series
89 +3 Ac Actinium 227.028
90 +4 Th Thorium 232.038
91 +5 Pa Protactinium 231.036
92 +6 U Uranium 238.029
93 +5 Np Neptunium 237.048
94 +4 Pu Plutonium 244.064
95 +3 Am Americium 243.061
96 +3 Cm Curium 247.070
97 +3 Bk Berkelium 247.070
98 +3 Cf Californium 251.080
99 +3 Es Einsteinium [254]
100 +3 Fm Fermium 257.095
101 +3 Md Mendelevium 258.1
102 +2 No Nobelium 259.101
103 +3 Lr Lawrencium [262]